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RADC-TDR-63-43

January 1963

FINAL TECHNICAL REPORT

CERAMIC-METAL SEALS FOR HIGH-POWER TUBES

Covering the Period
10 April 1961 through 31 October 1962

ELECTRONIC TUBE DIVISION
SPERRY GYROSCOPE COMPANY
DIVISION OF SPERRY RAND CORPORATION
GREAT NECK, NEW YORK

Sperry Report No. NA-8250-8331

Contract No. AF30(602)-2371

Prepared for

ROME AIR DEVELOPMENT CENTER
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
GRIFFISS AIR FORCE BASE, N.Y.

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Project No. 5573
Task No. 557303
Contract No. AF30(602)-2371

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FOREWORD

This final technical report discusses the metal-to-ceramic seal technology study conducted for the Rome Air Development Center, U.S. Air Force by the Electronic Tube Division, Sperry Gyroscope Company Division of Sperry Rand Corporation, Great Neck, New York. The work was performed under Contract No. AF30(602)-2371 during the period 10 April 1961 through 31 October 1962, and was a continuation of work performed by Sperry under Contract No. AF30(602)-2047. Four technical notes were published during the program. [1-4]*

The Techniques Section of the Electronic Tube Division was responsible for the entire program. The Sperry Materials Laboratory also participated, being responsible for the leak path study and the tensile test evaluation. In addition, this group designed and performed the mathematical calculations for statistical experiments 3 and 5. Sincere appreciation is expressed to Mr. Dirk Bussey of RADC and to the many other individuals whose contributions made this work possible.

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*References are given on page 47.

ABSTRACT

Variables associated with the fabrication of metal-to-ceramic seals were investigated to understand the mechanisms of failures and to improve manufacturing processes. The program primarily involved an extensive reliability study in which ceramic bodies, metallizing mixtures, plating, sintering, brazing, and other sealing practices were statistically evaluated. Environmental, life, and leak path studies supported the primary investigations. Recommended manufacturing procedures and controls were incorporated into a manual on metal-to-ceramic sealing techniques.

The results of the experiments were masked to a large extent by a residual error caused by uncontrolled and/or unknown variables. The ASTM test vehicle employed in the program was apparently the source of the variables.

An operating nonpumping ion gage was constructed. Further refinement of the device is suggested.

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SECTION I

INTRODUCTION

1-1. PURPOSE OF PROGRAM

During the previous program which Sperry conducted under Contract No. AF30(602)-2047, 200 metallizing compositions were evaluated to develop a ceramic-to-metal seal as strong as the ceramic member itself. Because of the wide excursions in the values obtained, it was deemed necessary to investigate the control parameters required to achieve reproducibility, an essential requirement for high-reliability structures. The current program consisted of two main efforts:

- An evaluation of those variables thought to be contributing to the wide fluctuations observed in metal-to-ceramic seal production operations, so that controls could be established to minimize these excursions from the mean.
- Employment of the necessary controls in a systematic evaluation of seals manufactured in accordance with established specifications.

The end objective of this program was the development of a metal-ceramic sealing manual describing in detail the practices and controls necessary to construct reliable metal-to-ceramic seals employed in the manufacture of highly reliable electronic tubes.

Three ceramic bodies ranging from 94- to 99.5-percent alumina were employed. A supporting program in which the causes of failure were evaluated was conducted so as to understand better the mechanisms of failure, and to improve further the over-all reliability of the recommended manufacturing procedures.

1-2. PHASES OF PROGRAM

The goals of this program were achieved through two main phases and five subsidiary phases supporting the main effort. These phases are as follows:

Phase I – Reliability Study – This phase, the principal effort of the program, consisted of a systematic evaluation of the variables associated with fabricating metal-to-ceramic seals. The work continued throughout the major portion of the program. Originally, six experiments were planned to evaluate eleven variables. The studies were combined into four experiments through the use of Latin-square statistically designed experiments which yielded more statistically significant information. The variables studied were thought, from previous work, to be those most significantly attributable to variations in seal strength. Late deliveries of the ceramic bodies and furnace breakdowns because of element burnouts caused a 3-month delay in the program.

Phase II – Manual – From the data obtained in Phase I, a manual of sealing technology for the manufacturing of reliable metal-to-ceramic hermetic seals was written. The manual describes in detail the recommended procedures, and the degree of controls and their relative importance in attaining reliability.

Phase III – Fabrication of Samples – During this phase, samples were fabricated on the basis of Phase I data for testing during Phases IV through VI. A minimum number of samples were fabricated because of the extended exploration of the many variables contributing to poor reproducibility.

Phase IV – Environmental Testing – Several manufacturing samples from the Sperry production facility were thermal cycled to evaluate their resistance to this environmental stress. The techniques employed were those described in the manual. The results indicated the degree of high reliability attained.

Phase V – Tube Compatibility Testing – The technical problems associated with the development of a working nonpumping ion gage, such as sputtering and d-c electrical leakage, shortened the test period. The successful completion of the construction of a nonpumping ion gage suggested the investigation of a similar existing metal-to-ceramic structure. Data are available on the generation of gasses as observed in similar metal-ceramic structures evaluated at Sperry. Information obtained under U.S. Army Signal Research and Development Laboratory Contract No. DA 36-039 SC-87389 is presented to indicate the level of dielectric loss in the metal-ceramic seal

areas. The extensive nature of the Signal Corps program, in which seals were evaluated in reference to r-f characteristics, provided more data than could be obtained during this present program.

Phase VI – Life Testing – This phase was to result in a number of nonpumping ion gages being sealed off under vacuum. These gages were to be periodically tested to indicate over-all reliability from a hermetic standpoint. The many technical problems encountered in the fabrication of a nonpumping gage shortened the available time for life testing. It is believed that additional funds would result in the production of a satisfactory nonpumping ion gage to fulfill this phase of the program. The gage could also be used for evaluating other characteristics, such as resistance to nuclear radiation, environmental resistance, and processing effects on final vacuum.

Phase VII – Leak Path Study – During this phase, the mechanisms of metal-ceramic seal leaks were investigated. One hundred and forty-one leaks in metal-ceramic seal assemblies were employed to develop techniques of tracing leak paths so that their basic causes could be evaluated. Several methods resulted in satisfactory tracking of the points of failure. A low-surface-tension low-viscosity penetrant dye was employed which, upon further treatment, resulted in a black carbon track that was visible in sectioned and polished samples. Many samples were observed and their causes of failure classified.

SECTION II

DISCUSSION

2-1. PHASE I – RELIABILITY STUDY

a. Variables Affecting Seal Strength

Phase I, the most important portion of the program, was originally planned such that eleven variables would be investigated. Previous work indicated that the following variables contributed most to variations in seal strength:

- Method of applying the metallizing (brush coating, spray, and roller application)
- Metallizing particle size
- Metallizing thickness
- Metallizing composition
- Sintering temperature
- Sintering time
- Sintering rate
- Impurity additions (additions to the metallizing composition in trace quantities as opposed to gross additions altering basic compositions)
- Sintering atmosphere dew point
- Sintering atmosphere hydrogen nitrogen ratio
- Type and thickness of electroplate on sintered metallizing layer.

The experiments were analyzed by a statistician familiar with metal-to-ceramic seals. As a result, the phase was changed so that there were four statistically designed experiments following the organization of Latin squares. This type of experimental design enables extraction of more useful information from a similar number of experimental tests. In addition, the effects of interactions of the variables can be determined, and a measure of the degree of statistical significance can be assigned to these variations.

Eitel-McCullough reported that high seal reliability was obtained by controlling the uniformity of the metallizing composition by roller-coat application. [5] Spurck, et al reported a decrease in tensile test range from ±40 to ±20 percent, with the same mean of 10,000 psi, through the use of a uniformly thick metallizing tape. [6] Sperry has observed that the ASTM sample strength varies for hand-painted metallizing samples, depending upon whether trained operators or untrained operators apply the metallizing mix. [7] These variations were as high as 50 percent.

Particle size is thought to contribute to the ease and rate of sintering. Sintering of two particles has been shown to occur by two mechanisms. [8] The particles tend to attach at their contact points by the development of a lens. This lens becomes larger by the vapor transport of particle material to the lens area. Also, the lens area acts as a source of lattice vacancies which tend to migrate to the main body of the particle. Both mechanisms are accelerated as particle size decreases.

The effect of thickness of the metallizing coating has been observed to cause considerable variations in seal strength. Unevenly metallized seals, when destructively tested, show variations in seal strength by the selective pulling of ceramic in areas of specific thicknesses. The different thicknesses of the metallizing apparently contribute to the over-all reliability of the seal. [9]

It was shown in the previous study, conducted under Contract No. AF30(602)-2047, that the composition of the mixes employed with a variety of ceramics contributes greatly to the ultimate seal strength and reliability. Several of the mixes developed in the previous program were employed for the present study. Temperature, time, and rate of sintering, as well as impurity additions, were also demonstrated in the previous program to contribute significantly to over-all seal strength. Furnace atmosphere conditions of dew point and gas compositions affect the oxide balance in the solid-state and liquid reactions of the sintering operations, and should contribute to a large extent in the over-all reliability and reproducibility of these systems. The type and thickness of electroplating employed could also contribute to seal reliability because of the residual stresses established in the plated layers.

b. Experiment 1

A preliminary experiment was conducted to establish the type of plating, plating thickness, and the brazing schedule which would be employed in the evaluation of all the other variables. The experiment was designed as shown in table 1; the resultant raw data are listed in table 2.* An analysis of variance was performed on these raw data to establish the degree each variable contributed to the over-all experiment.

The ability to detect, measure, and assign relative levels of importance to variations observed is dependent upon the residual error of the experiment. Residual error is defined as the variation caused by uncontrolled and/or unknown variables acting upon the experimental variables being tested. The greater the effect of these unknown variables, the less sensitive the experiment is to the variables under study. "F ratio tables" are available which indicate the statistical significance of the variables being studied. [10] The F ratio is the proportion of variation of a variable under study to the residual error. The higher the F ratio, the more confidence may be placed on the results of the experiment. Therefore, as the residual error decreases, the F ratio increases for a given measured variance.

In Experiment 1, the residual error was rather high; thus, the sensitivity of the over-all experiment was decreased in assigning significance to the variables studied. The only variable which could be assigned any statistical significance was the difference between the two ceramic bodies employed. This significance was at a 1-percent level, which means that the possibility of this difference being due to chance is 1 in 100. For a significance of 0.1 percent, or 1 in 1000, attributable to chance, the F ratio would have had to be 12 or higher. Thus, it can be seen that the residual error greatly decreased the sensitivity of the over-all experiment. Close visual examination revealed some scattering effect in the type of breaks, suggesting the test vehicle as the source of residual error.

Three minor variables--plating bath differences, interaction between plating and brazing, and interaction between plating thickness and the type of ceramic--had a degree of significance less than 5 percent. If the residual error decreased,

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*Tables are in numerical order starting on page 49.

these variables may have shown a statistical significance. The 32-sample average for all conditions employing AD94 was 9671 psi, whereas the average of 32 AL300 samples was 8331 psi. An apparent degradation of averages of the four replicates in this experiment suggested that time degradation was acting, but further examination of other tests did not substantiate this.

Suspecting the test vehicle as the source of the residual error, a set of conditions for plating bath, plating thickness, and brazing cycle was selected for all ensuing experiments; these variables lacked any statistical significance in the first experiment. The conditions were (1) sulphamate nickel electroplating bath, (2) plating thickness of 0.0002 inch, and (3) brazing cycle of 10 minutes preheat, 15 minutes braze, and two 10-minute post-cool subcycles. Sulphamate nickel was selected instead of ammonium nickel because of its better throwing power, resultant uniform buildup, and faster plating rate. The 0.0002-inch plate was more economical from a time standpoint when compared to a double-thickness plating study. Also, the brazing cycle was selected on basis of time economy.

c. Experiment 2

The following variables were studied in experiment 2 (see table 3):

- Two ceramics -- AD-94 and AL-300
- Three methods of application -- hand, roller, and spray coating
- Three ball-milling times -- 2, 5, and 10 days
- Two metallizing layer thicknesses -- 0.0006 and 0.0012 inch.

The milling times were selected on the basis of an experiment which determined the average particle size versus milling times. Figure 1 illustrates that 2, 5, and 10 days gave average particle sizes of approximately 2, 1.2, and 0.7 microns, respectively. Originally, the variables were to be replicated six times, but this was reduced to three because of a furnace failure after the third replicate. Mix 65B was employed with both ceramics.

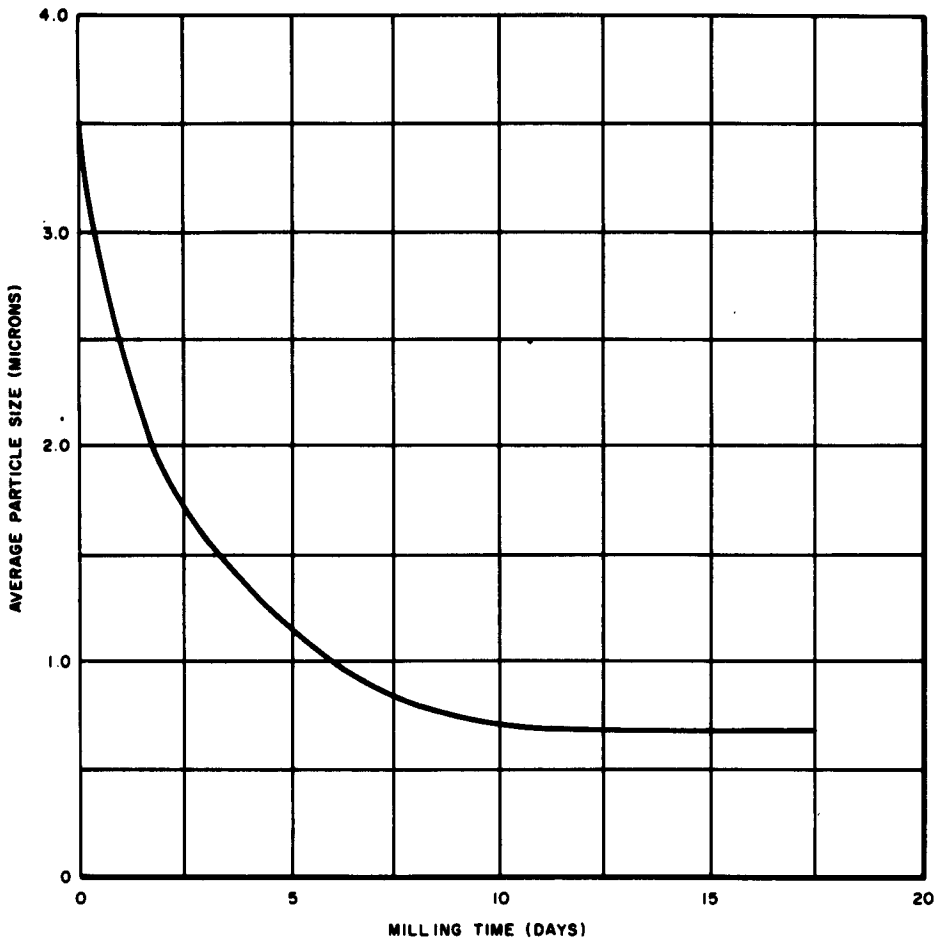


FIGURE 1. PARTICLE SIZE VS MILLING TIME

The spray-, roller-, and hand-coating equipment are illustrated in figures 2, 3, and 4, respectively. Preliminary evaluations of the spray- and roller-coating machines were made so as to become familiar with their operation. This was essential so that a fair comparison with hand-painting could be made, as this was developed to a high degree at Sperry. The minimum thickness of 0.0006 inch uniformly reproduced by the roller-coating machine determined the single-layer thickness employed in all three application techniques. The two-coat layer employed two similar layer thicknesses, each separately sintered.

Modifications to the high-temperature sintering furnace, illustrated in figure 5, enabled temperatures to be maintained and reproduced to within ± 1°C. Also, modifications of the wetting bottle, providing insulation to the leads entering the furnace, enabled dew points greater than ambient room temperature to be maintained for high dew-point applications. The Baldwin tensile tester, shown in figure 6, was modified to allow better alignment of the tensile pulling fixtures. Possibilities for misalignment existed in the tensiling grips and the specimen holders, allowing slippage of the specimens which resulted in unwanted sheer forces.

A strain-gage analysis was performed on a metallic replica of a brazed ASTM sample, resulting in a high degree of improvement in the over-all tensile testing of the samples. Figure 7 illustrates the improvement in the uniformity of stresses measured at four 90-degree intervals.

The uniformity of stress at the periphery of the seal area indicates the over-all decrease of actual stresses resulting in sheer stress applied to the samples. This decrease was from 6180 psi to 1240 psi. The modified specimen holder with the strain gage and aluminum sample in position is shown in figure 8. This improvement of the tensile testing holder resulted in a decrease of the residual error of the experiment, thereby making future tests more sensitive to the variables under study, and minimizing the effects of unknown variables acting upon the experiment.

The raw data of experiment 2 is listed in table 4. An analysis of variance was performed on the tensile values; the ceramics again showed a significant difference statistically. The significance was at the 5-percent level, which indicated a

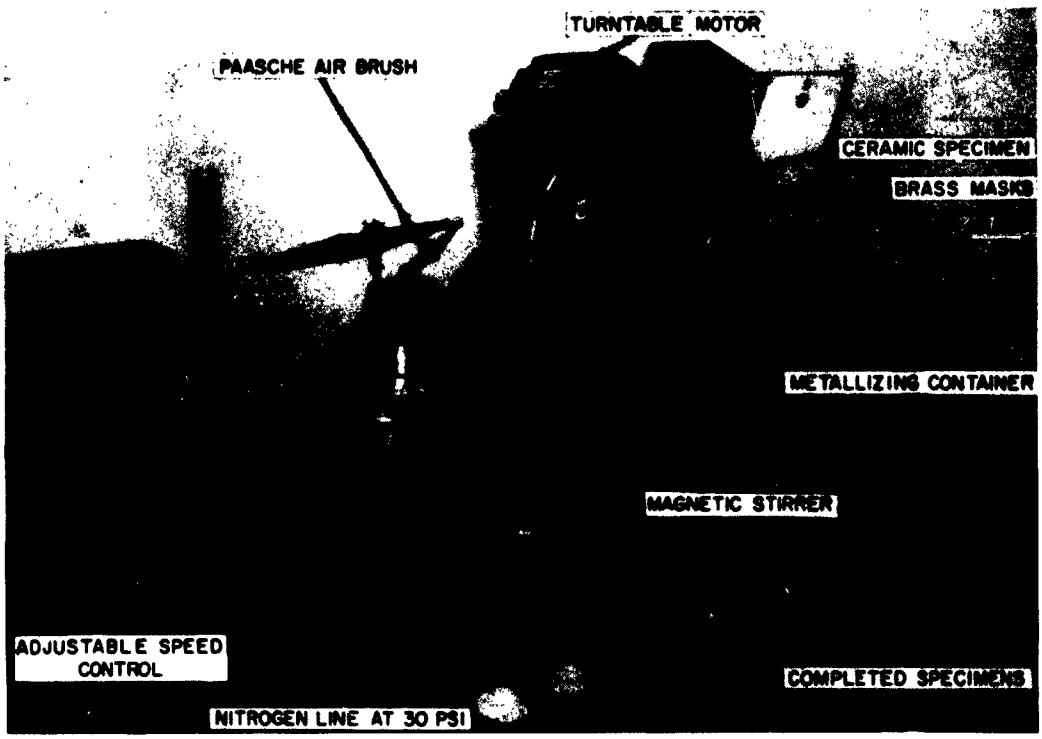


FIGURE 2. SPRAY-COATING EQUIPMENT

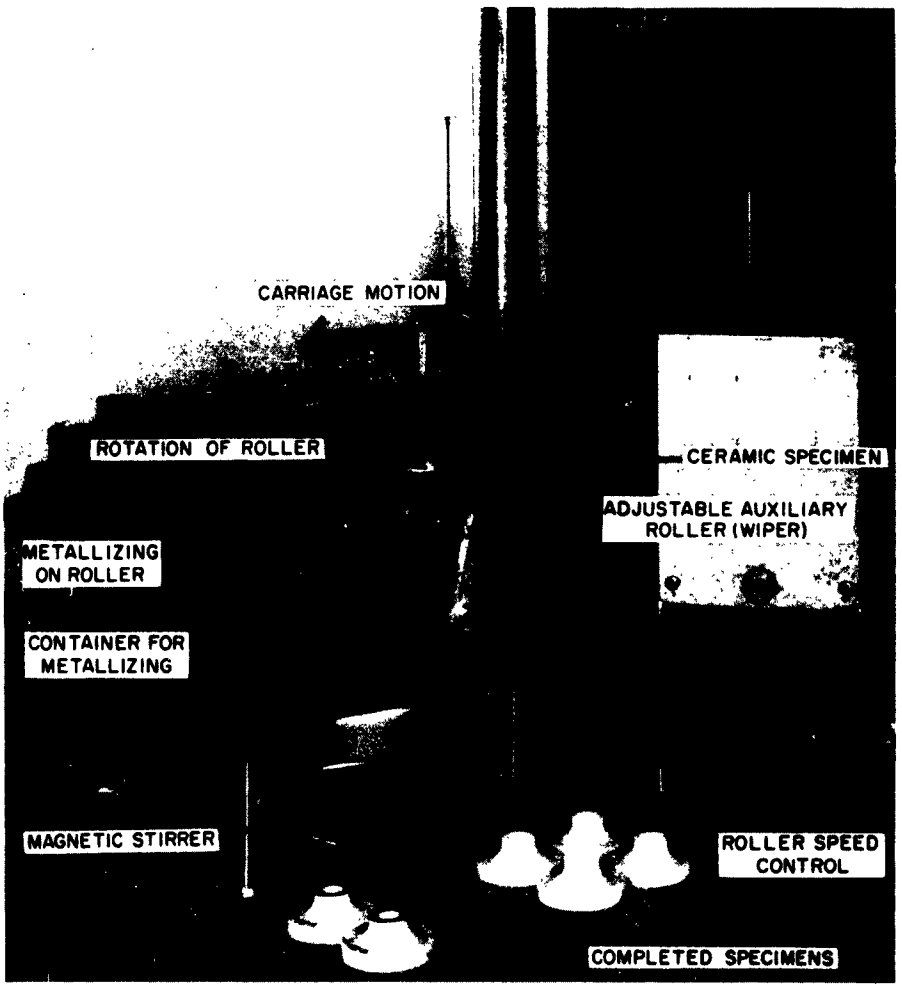


FIGURE 3. ROLLER-COATING EQUIPMENT

FIGURE 4. HAND-COATING FACILITY

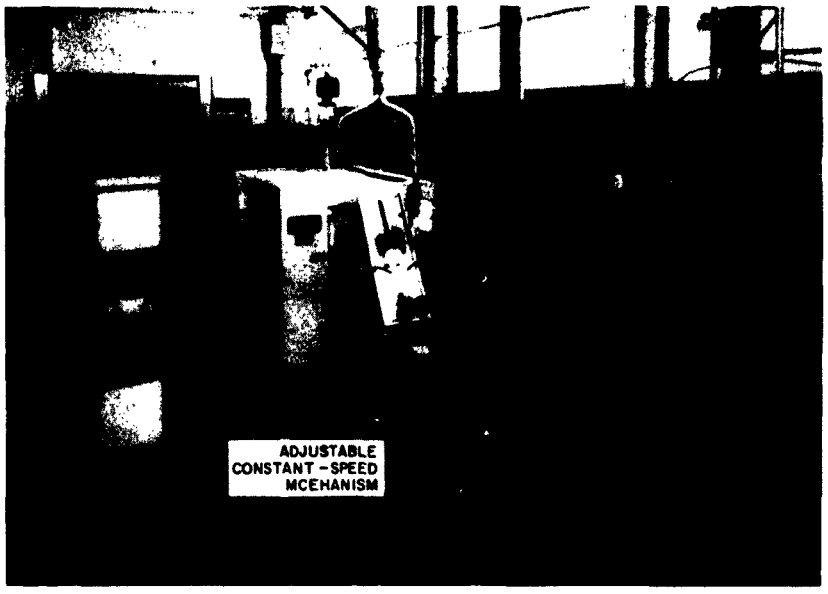


FIGURE 5. HIGH-TEMPERATURE SINTERING FURNACE

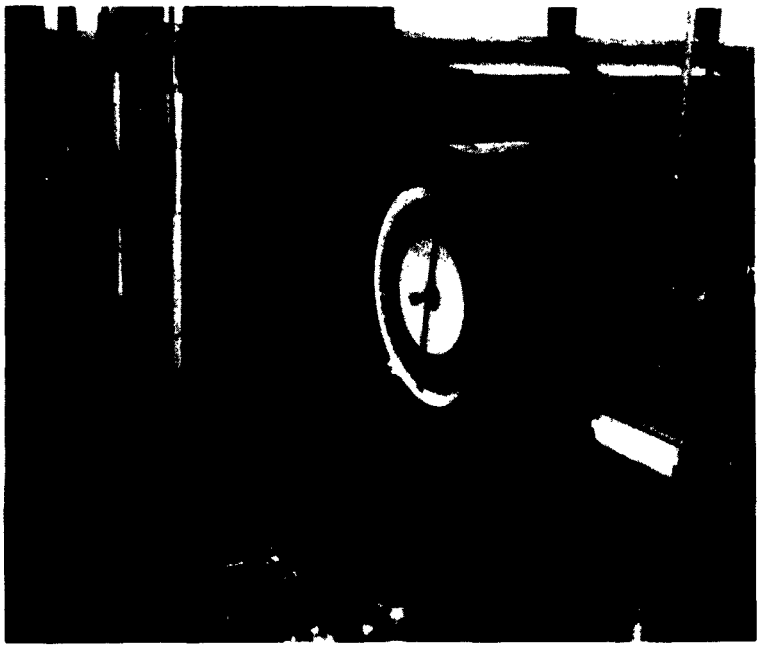


FIGURE 6. BALDWIN TENSILE-TESTING EQUIPMENT

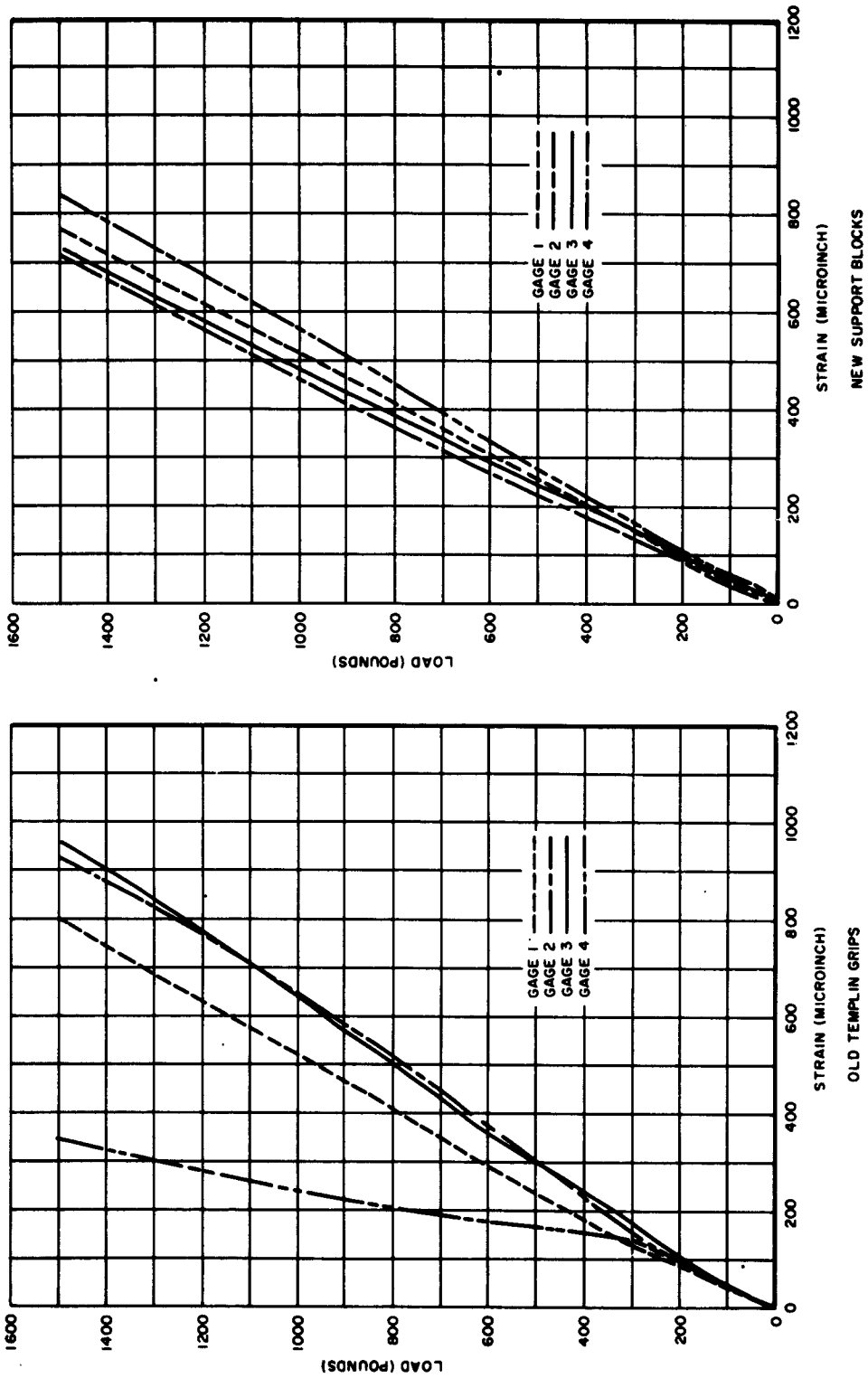


FIGURE 7. ASTM SPECIMEN HOLDER STRESS MEASUREMENTS

higher degree of residual error in this experiment than in experiment 1, in which there was a 1-percent level. The number of metallizing layers was significant at the 1-percent level. The interaction of the ceramic, the method of application, and the number of layers were significant to the 0.1-percent level, indicating that the difference could be attributable to these variables with the probability of chance being 1 in 1000.

Main variables cannot be tested against the residual for the entire experiment when an interaction shows up as significant. Neither the lower-order interactions (variables taken two at a time constitute first-order interaction and variables taken three at a time constitute second-order interaction) nor the main variables may be tested; therefore, it is necessary to perform separate analyses, as shown in tables 5 and 6 for one and two-layer samples. In the one-layer analysis of variance, no main variables or interactions proved significant. This was even true of the two ceramics. The two-layer samples proved significant at the 0.1-percent level in the interaction of the ceramic and the method of applying the metallizing.

The ceramics also showed a significant difference; but once again, when there is a significance in an interaction, a further breakdown analysis is indicated. This could have been made by performing separate analyses on the two ceramics, rather than on the number of layers. The fact that nothing was significant in the one-layer samples and that two variables showed significant differences in the two-layer samples proves that the layers themselves are significantly different. Both experiments 1 and 2 showed the AL300 and AD94 ceramics to be significantly different. Table 7 indicates the average strength of the metallizing bond for all conditions of each ceramic in experiments 1 and 2.

Variations due to milling time were well within the error of measurement. The 2-day ball-milling tensile average of all samples in experiment 2 was 11,034 psi, the 5-day tensile average was 11,269 psi, and the 10-day tensile average was 10,919 psi, all within 300 psi. Although there was an interaction between ceramics and the method of application (significant in the two-layer breakdown analysis), nothing was planned to investigate this further because of time limitations. The difference between the one-layer and two-layer samples was significant at the 1-percent level; however, the spread between the two averages was not very great -- one-layer samples averaged 10,494 psi and two-layer samples averaged 11,653 psi.

The conclusions drawn from experiments 1 and 2 suffered from one common shortcoming -- the replication error was exceedingly high for this type of work. Closer examination of the samples from the two experiments indicated a wide variety in the type of fractures during the testing phase, which do not appear in metal-to-ceramic seal breaks normally tested in routine Sperry process controls. These variations further supported the suspicions that the ceramic-to-ceramic brazed ASTM sample was the primary source of this uncontrolled variable which masked many of the tests performed.

Experiments 1 and 2 had over-all coefficients of variation of 22 percent and 25 percent, respectively. This lack of good replication is further emphasized in comparing the over-all averages for both experiments, experiments 1 and 2 being 9001 and 11,074 psi, respectively. Althoug the same ceramics and mixes were employed in both experiments, uncontrolled or unknown variables acting upon these tests accounted for the 2000-psi increase in strength. Throughout all of this work, many instances of time dependance became apparent--the time between cleaning the ceramics and applying metallizing, between metallizing application and sintering, between sintering and plating, and between plating and brazing.

d. Experiment 4

Prior to conducting experiment 3 (see paragraph 2-1e), a small time study, designated experiment 4, was executed. The design of experiment 4 is listed in table 8, and the performance schedule is listed in table 9. With single-layer metallized AL300 ceramic, the average tensile strength was 7,570 psi, with a coefficient of variation of 23.5 percent. The coefficient of variation was high, as in experiments 1 and 2, but the unexplained severe drop in tensile strength could not be accounted for from the data recorded.

An analysis of variance was performed, with no main variables or interactions showing any significance. The sensitivity of this experiment was masked by the high residual error. One possible explanation of the marked decrease in average seal strength is the method of plating. In experiment 2, a plating rack was employed for minimum contact points for the electroplating of the sintered layer of metallizing; in experiment 4, because of its limited size, the few pieces of metallized ceramic were wired

for plating, producing a shielding or masking over the ceramic and minimizing the amount of plating in this particular area. These sites could then be points of uneven stress or severely etched metallizing. No significance could be attributed to time delays in the areas studied, because all of the samples were plated with the same wire technique. The ceramic-to-ceramic brazed ASTM sample appears to have caused severe stresses in the finished joints, which could possibly create abnormally sensitive conditions for additional stresses caused by the above mentioned problems.

e. Experiment 3

Experiment 3, the most complex study of the program, employed 3 ceramics and 13 mixes, for a combined total of 16 different selected mix-ceramic combinations. Each combination was then sintered at three temperatures--1425, 1500, and 1575°C--and at three heating schedules--4, 6, and 8 hours. The 16 mix-ceramic interactions, 3 temperatures, and 3 heating schedules total 144 conditions, which were replicated 4 times for a total of 576 samples. Table 10 shows the design of the experiment, and table 11 lists the mix compositions.

The raw data for the three ceramic bodies--AD94, AL300 and AD995--are listed in tables 12, 13, and 14, along with their averages for all temperatures and all cycles, and the grand average. As observed in other experiments and in production operations at Sperry, seal strength is inversely proportional to the percent alumina content. The AD94, a 94-percent alumina body, had an average for all conditions of 10, 217 psi; the AL300 containing 96-percent alumina had an over-all average for all conditions of 8887 psi; the 99. 5-percent AD995 alumina had an over-all average of 5686 psi. The averages of the raw data can be presented in a more useful fashion as illustrated in table 15, in which the sensitivity to soak time and sintering temperature is more readily discerned. These data include tensile averages and coefficients of variation for all samples. To illustrate the effects of temperature and/or sintering time, table 16 combines all samples for each of the conditions. For example, ceramic-mix combination 1 under 1425°C and all cycles contains the 4-, 6-, and 8-hour cycle samples (total of 12 samples) for a grand average of 10, 111 psi; the standard deviation is 1529 psi and the coefficient of variation is 15. 1 percent.

To illustrate the lack of time dependence upon these experiments, the values are listed in the order of sintering and coating, as shown in table 17. Table 18 illustrates the sum total of all samples sintered at each temperature and cycle for each ceramic. The sum total of all ceramics and mix combinations for the specific times and temperatures indicated is given in table 19. Each condition is represented by an average of 64 samples; the averages across and down for total temperatures and total times, respectively, are for 192 samples each. The grand total of 8869 psi, with a standard deviation of 3260 psi, yielded a 36.76-percent coefficient of variation, higher than that for experiments 1, 2, and 4. The greater number of variables and the greater number of operations permits larger residual errors, which make the experiment insensitive to many of the studied variables. The inclusion of the high-alumina AD995 further added to the prestressing condition of the ceramic-to-ceramic seal, probably causing the over-all experiment to be more sensitive to minor variations in processing.

Tables 20, 21, and 22 are the analyses of variance on AD94, AL300, and AD995, respectively. The F ratio indicated that the interaction between mixes and ceramics is significant for AD94 and AL300 at the 0.1-percent level; but this was to be expected from previous data which indicated a large difference in the coefficient of variation between mixes 65 and 91 for AD94 and mix 72 and 65 for AL300. The AD995 ceramic was insensitive to the three mixes employed in this test. The mix and heat-cycle interaction for AD94 was statistically significant at the 5-percent level; but again with a primary variation significant, a further breakdown study must be conducted in order to verify the interactions. For the AL300 ceramic, there was a 5-percent statistical significance to heating cycles which is subject to a breakdown analysis because of the 0.1-percent significance for the sintering temperatures and a 5-percent significance for the mixes and sintering temperature interaction. All results were somewhat expected, and can be explained by the type of ceramic body and the mix employed in each case.

Table 15 shows that AD94 in conjunction with mixes 65A, 65B, and 65C--ceramic-mix combinations 1, 2, and 3, respectively--displays little dependence upon sintering temperature or time. The active titanium metal in the mixes can account for the lack of interaction under these conditions. The active nature of

the mixes suggests the reaction had gone to completion before any of these conditions had been reached, and any further processing during the sintering operation is superfluous. Samples 10, 11, and 12- AL300 employed with mixes 65A, 65B and 65C--show a marked difference in response to sintering temperatures and cycles. Although the scatter of tensile values from one sintering temperature to another and from one sintering cycle to another for the same ceramic-mix combination can be explained as being due to the experimental error, the variations from mix to mix can be accounted for by the decrease in active metal content in going from sample 10 to 12. This 96-percent alumina body had less glassy phase to react with the active metal; therefore,more active metal is required to bring about a final reaction so that the concentration at the surface boundary can be maintained at the high level required for penetration. The decrease in average strength for the entire experiment is indicative of a mechanism of this type. The high coefficient of variation (in the thirties) for the high-alumina AL300, as compared to the markedly lower coefficient of variation (in the low twenties) for the low-alumina AD94, further supports the contention of the active metal reacting with the glassy phase.

The sensitivity of AD94 to the variations in the minor constituents of the molybdenum mix can be understood by comparing samples 4, 5, and 6 with samples 1, 2, and 3; the values are well within the experimental error. A sharp decrease in the percent coefficient of variation for the molybdenum plua lithium manganate mix in samples 4, 5, and 6 can be explained by the mobility of the lithium manganate before decomposition. Other tests and manufacturing experience with this mix indicate that the material tends to bleed or penetrate the unsintered molybdenum matrix before decomposition to metallic molybdenum occurs. This provides more material at the interface of the ceramic and the mix for reaction to occur. Exceptional uniformity of the three samples indicates that a lower coefficient of variation may be possible with proper application of the mix, which would result in a more intimate attachment and denser application of the unsintered mix.

Samples 7, 8, and 9--the AL300 with the calcium oxide glass-maker addition--produced the worst scatter and the highest coefficient of variation of any of the samples. The average values are once again in the same general area, indicating little effect

in seal strength from changing the over-all composition; however, the decrease in the coefficient of variation, with a decrease in calcium oxide content, suggests that this material is very sensitive to sintering conditions. Samples 13, 14, and 15--the samples with 99.5-percent AD995--demonstrated low seal strength and high variations, or poor control. Here again the glass and manganese content did not appear to affect the relative properties of the sintered mix. Sample 16, made of AD94 and the standard Sperry mix, exhibited values in the same range as the other AD94 samples within the experimental error, the coefficient of variation being in the same order of magnitude.

The scattering in seal strength averages and coefficients of variation of the various ceramic-mix combinations and sintering conditions prevents clear-cut conclusions to be drawn from the data. For low-purity aluminas (those in the 94-percent range), it can be generalized that sintering time and temperature are somewhat unimportant in attaining good seal strength and reproducibility. As the alumina content increases to 96 and 99.5 percent, the minimum sintering temperature of 1425°C becomes exceedingly marginal, causing very large variations and poor seal strengths. Increasing the temperature to 1500 and 1575°C produces better seal strength, on the average, and lower coefficients of variation. The length of sintering appears to play a minor role for all ceramics and all bodies, with the coefficient of variation increasing with the alumina content. The higher the alumina content, the more sensitive would be the tests to a nonuniform expansion characteristic of the mating ASTM pieces, causing prestress conditions before tensile test applications.

The above discussion indicates the necessity for treating each mix-ceramic combination as a separate system to be studied for maximum control purposes. Although certain combinations gave optimum results of tensile strength and coefficient of variation, within each category of mix and ceramic the results were roughly consistent. Continued use of these combinations will enable the user to familiarize himself with the particular peculiarities of each system.

f. Experiment 5

Experiment 5 was established to investigate the problems of reproducibility and to obtain a better understanding of the residual error of the previous experiments. This experiment was designed to test some of the observed causes of variation in the

previous experiments, and to establish a level of reproducibility for a long-term run of the samples (see table 23).

The three ceramics used in experiment 3 were also employed in experiment 5. Also, the mixes found to be optimum from experiment 3 were used. Additives of 0.5 percent iron and 0.5 percent silica were added to determine the effects of ball-mill impurities on the mixes. Both ball-milled and colloid-milled mixes were employed to study the variations due to different methods of preparing the mix. Thus, 3 ceramics, with 2 mixes for each, and 3 conditions of additives total 18 different conditions. For each set of 18 conditions, 32 tensile halves were run once a week for 14 weeks to study the life of the mixes and the effects, if any, of the additives or colloid milling on the deterioration of the mix.

Table 24 illustrates the raw data for the 14 replicates of experiment 5. Sample 7 was run just before vacation; the operator who had been hand-painting the samples for the first 6 weeks was unavailable. Another operator, untrained in painting the metallizing mix, applied what visually appeared to be the same coating. The other operations of sintering, electroplating, brazing, and testing were performed by the personnel employed in the previous tests. There was a marked decrease in the over-all sample tensile values and an approximately 60-percent decrease in the average for the 18 samples, illustrating the influence of the operator on seal strength.

It had been observed that tooling for alignment of the two mating halves of ceramic ASTM samples created some degree of eccentricity. The brazing fixture, through sample 7, had consisted of a greened stainless-steel pin inserted in a greened stainless-steel base, onto which the two mating halves with their solder washer were placed. A 1-pound weight of greened stainless steel was placed on top of this combination to ensure intimate contact between the plated metallized surfaces and the brazing ring. An evaluation of the problem revealed that the allowance of the clearance for fit at the maximum temperature enabled the pieces to move slightly before the solder froze during the cooling cycle.

An attempt was made to employ a split column, as illustrated in figure 9. However, the difficulties in constructing a split mandrel which would operate at a copper-brazing heat

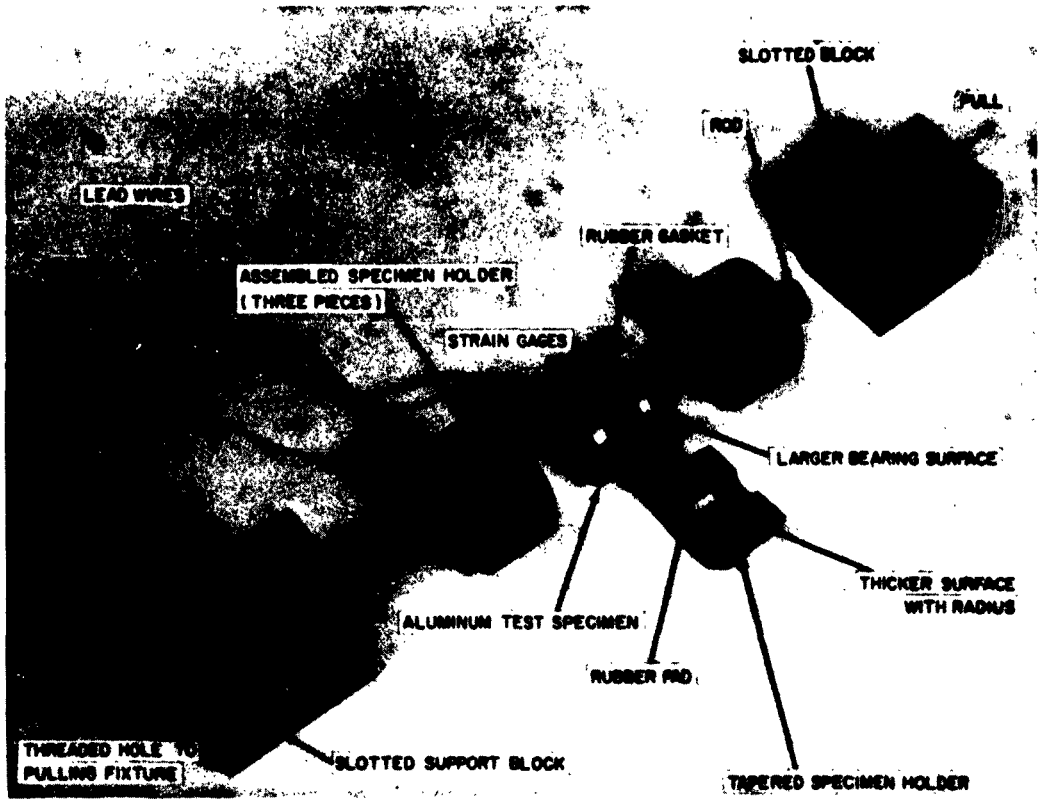


FIGURE 8. MODIFIED SPECIMEN HOLDER AND ALUMINUM ASTM SPECIMEN

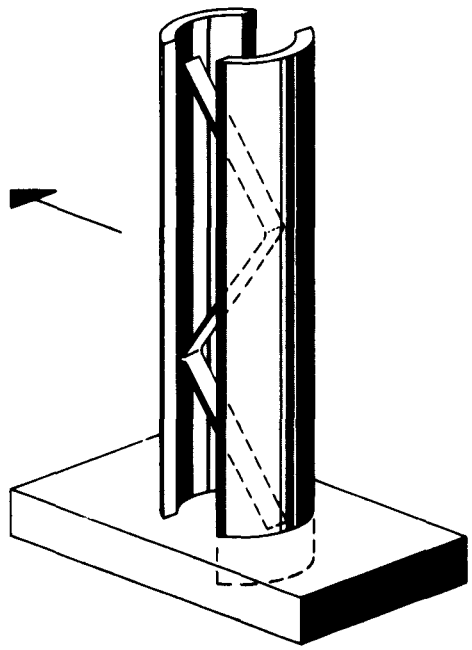


FIGURE 9. SPLIT BRAZING FIXTURE

of 1150°C appeared somewhat difficult. A second fix was effected through the use of a carbon fixture which was machined from dense graphite and physically similar to the greened stainless-steel fixture. The thermal mismatch at brazing heat was much less and the resultant alignment greater than in the first design in which a snug mandrel made of steel would crack any samples slightly undersized on the inside dimension. The carbon fixture at brazing temperature was more compliant and did not crack the samples. Samples 8 through 14 employed the carbon fixture. Sample 11 was a reoccurrence of the situation of sample 7, in which the inexperienced operator applied the metallizing. Again, large variations from the results of inexperienced personnel were reflected in the seal strength.

Table 25 indicates the measured eccentricities of the samples and illustrates the improvement in alignment due to the use of the carbon fixture. The values are listed in filar units on the microscope field, with a 0.66 objective and a 20x ocular. One filar unit is calculated to be 0.0003 inch. Arranging the averages of all samples, but rejecting samples 7 and 11 because of the variations introduced by an inexperienced operator, it can be seen that the coefficient of variation of the carbon fixture was slightly better than that for the metal fixture. Table 26 lists the summation of all samples for each run, tensile average, percent coefficient of variation, and average eccentricity. There is no apparent correlation between eccentricity and average seal strength or coefficient of variation.

A final treatment of the data is listed in table 27, which is an analysis of the variance of the experiment. The analysis was performed on the first six and the last six sets of samples, as if two separate experiments had been run independently. The residual error with the metal fixture is slightly larger than that with the carbon fixture, and tends to mask the detectable significances to a larger extent. This result further supports the contention that the ceramic-to-ceramic ASTM samples are more sensitive to minor variations in seal techniques than are the normally encountered metal-to-ceramic seals. The metal member in metal-to-ceramic seals is somewhat more compliant than the ceramic, and thus takes up the differential expansion mismatch and minimizes the prestressed condition of the brazed ceramic-metal joints.

The mix milling method for AD995 approaches statistical significance, but nothing in the first six sets can be designated as such. With the carbon fixture, however, the statistical significances of the 5-percent level for the impurity with AL300, and the milling technique of the mixes with AD94 are noted. The coefficients of variation for these experiments are in the same order of magnitude as all previous work, indicating large residual errors which tend to mask significances unless they are profound in their influence on the tensile strength.

The over-all results of this program show more negative than positive indications of variables attributable to variations in seal strength. While this is valuable in the sense that the negative indications will not require close control in seal fabrication, the following contributions encountered under various conditions in these experiments contributed to variation of seal strength and reproducibility:

- Composition of the ceramic body
- Thickness of the metallized layer
- Method of application
- Composition of metallizing
- Sintering temperature
- Sintering cycle
- Impurities in mix
- Particle size of metallizing mix.

These attributes were not significant under all circumstances of type of ceramic body and/or metallizing mixture composition. This is thought to be due to the variety of mechanisms involved in the sealing of various ceramic-mix combinations employing molybdenum with no additions, molybdenum with active metals, and molybdenum with glass-forming materials or glass itself. The final interpretation and use of these data are discussed in paragraph 2-2.

2-2. PHASE II - SEALING MANUAL

At the beginning of the series of experiments, the intent was to develop a scientific procedure for the reproducible production of reliable metal-to-ceramic vacuum-tight seals.

Throughout the quarterly technical notes[1-4] the one serious problem encountered--the large residual error--has been apparent. All of the experiments, without exception, suffered from this inexplicable shortcoming. All attempts to replicate supposedly identical conditions failed, with the result being a decrease or complete lack of faith in any indicated differences. Although some significances were observed at the 5-, 1-, and 0.1-percent levels, the underlying fact of uncontrolled variables acting throughout the experiments leaves one reluctant to accept the results with any finality.

The evaluation of these experiments with a test vehicle composed of a metal-to-ceramic seal assembly would possibly have given more indication of the real condition found in metal-to-ceramic seals. The sensitivity of the prestressed ceramic-to-ceramic assemblies to processing variations may have been disproportionate, causing the large residual errors observed.

Several trends and variations were noted throughout the experiments, and these can be capitalized upon by treating them as trends rather than as strict rules to be followed. Most of the tests were run under strict laboratory-controlled conditions employing control techniques that are more rigid than normally encountered in production facilities (such as the temperature control of the sintering furnace, the plating thickness control, and the brazing cycles and temperatures). The above suggests that a reproducible process under production conditions is doubtful. However, as previously stated, reliability can be attained by reducing the coefficient of variation of an average tensile strength in the acceptable range, or by raising all of the tensile values much higher so that the lowest value is still acceptable.

Until recently, metallizing and metal-to-ceramic sealing was classified as a skilled craft which relied heavily upon the developed techniques of the operators in applying the metallizing, judging the brazing cycles, and carefully fixturing and placing the solder rings. Many companies are presently engaged in manufacturing reliable metal-to-ceramic electronic tubes. Their apparent means of attaining high reliability has been in maintaining a high enough tensile average so as to include the lowest values of the spread within the acceptable range. Many variables have been studied in an attempt to determine a relative importance in the determination of seal strength, but many

more unknown variables remain to be accounted for. Further work in the development of a representative test vehicle would greatly aid in the true representation of the many and varied sealing problems known to exist. Further insight into the possible causes of variation are discovered continuously in the routine production techniques being developed over the years. It is with this process in mind that the sealing manual was written--not as a precise tool for the exact, reproducible production of seals, but rather as a guide for both the production man and the newcomer to the field of metal-to-ceramic sealing.

For the fabrication of metal-to-ceramic seal structures, it is advisable to select a material on the basis of its mechanical, electrical, and other physical properties which are suitable for most or all applications. Once the ceramic has been selected (or possibly several ceramics for varied applications, such as r-f windows and base insulators), these materials should then be employed in a given set of facilities to determine the peculiarities of the combination. Through usage, insight is obtained into the limitations and possibilities of the combination of ceramic and facilities. Changing any portion of the facilities necessitates a new evaluation of the over-all system. Therefore, it is recommended that as much of the facility as possible be reserved and allocated for the sole use of the ceramic operation. This includes the areas and equipment for cleaning, air-firing, metallizing, sintering, electroplating, and assembly; the fixtures and jigs; the brazing furnaces; and other supporting operations. Many tools for measuring and monitoring the various processes involved are available and should be employed where practical.

2-3. PHASE III - FABRICATION OF SAMPLES

Early in the program, it was decided to use nonpumping ion-gage assemblies, described in paragraph 2-6 (Phase VI), to evaluate the seals under environmental testing conditions and to evaluate the by-products of the seals for tube compatibility testing (as listed in paragraphs 2-4 and 2-5, respectively). Many difficulties were encountered in the development and fabrication of a nonpumping ion gage, and time did not permit the fabrication of samples for this phase, although a successful operating gage was fabricated before the close of the program. The ceramic and metal members were obtained, but a lack of

funds and time prevented the construction of the 90 gages--30 each of AD94, AL300, and AD995 as originally planned.

2-4. PHASE IV - ENVIRONMENTAL TESTING

Because of the time expended in obtaining a nonpumping ion gage, none of these devices was available for the planned environmental stress-resistance tests. In lieu of information from this vigorous testing, data are presented which illustrate typical samples environmentally tested at Sperry in the quality control of the metal-ceramic sealing facility. The following list indicates the conditions and the number of cycles to which several assemblies have been subjected:

Quantity	Thermal Cycle	Assembly	% Yield
36	650°C in air	Cone windows (approximately 1.5-inch diameter)	98 .
2	650°C in forming gas (9 cycles)	Windows (8.25-inch diameter)	100
24	650°C in forming gas	Coaxial Antennas (approximately 0.25-inch OD)	100
11	650°C in forming gas	Base insulators (12-inch diameter)	91

In most cases, if a seal succeeded in passing the first thermal cycle, its life on successive cycles was of a high order of magnitude; many tests were discontinued for lack of time.

Several instances can be cited in which selected samples were continued to the point of catastrophic failure as determined by helium mass spectrometer leaks. Small coaxial antenna seals of 0.100 inch in diameter, and high-alumina windows 0.060-inch thick and 1.617 inches in diameter, copper brazed in Kovar and/or silver-copper eutectic brazed in copper sleeves, have exceeded 20 such cycles. Other more rigorous tests, such as thermal shock, have been applied to metal-ceramic assemblies; the window assembly shown in figure 10 was heated to 650°C in an air furnace and suddenly removed to room temperature. The

metal members were gold-plated to minimize or eliminate oxidation during the air-firing. The brazing cycles employed in joining windows and coaxial antennas to the metal members of tube assemblies present even greater stresses for environmental evaluations. For an antenna which is normally copper brazed with a concentrator-modified induction heater, the braze is effected at the joint; the seal area 1 inch away rarely attains a temperature higher than 100°C. Mechanical stresses induced in the sleeve are transmitted to the seal area. Antennas of this type have been successfully employed for several years in highly reliable Sperry products.

2-5. PHASE V - TUBE COMPATIBILITY TESTING

This phase of the program was also dependent upon the construction of nonpumping gages, which were to have been analyzed during bakeout for residual gases. This portion of the program was not conducted with these gages, but the data in the following list illustrate the types of gases and their relative concentrations observed during the 200°C bakeout of metal-ceramic tube assemblies having ceramics and brazing materials similar to those the gages contained:

Gas	Relative Concentration
Water Vapor	65
Carbon Monoxide	15
Acetone	<< 1

Although most of these gases can be attributed to the outgassing of the surface of the metal members of the tube, no gases harmful or poisonous to the cathode were observed.

Evaluation of the r-f properties of metal-ceramic seals presented a problem in that the equipment and monies required were not available for this program. A program administered by the U.S. Army Signal Research and Development Laboratory to investigate window waveguide failure mechanisms was being simultaneously conducted at Sperry. The following observations from that program relate to metal-ceramic seals:[11]

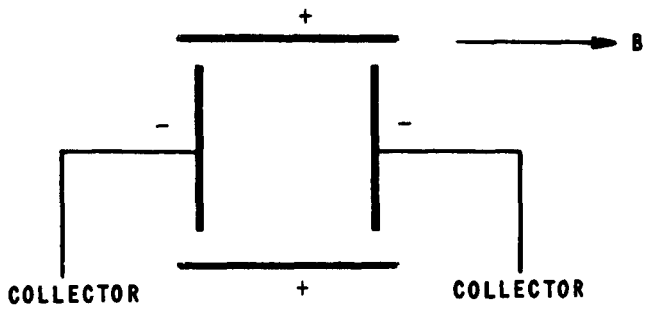
> Arcing at the window seal is a serious problem that requires careful attention to fabrication techniques... A large fillet of brazing material forms a strong seal,

but it presents a sharp edge to the tangential electric field on the window surface. By using a compliant waveguide wall, a tight seal can be formed with a minimum of brazing material confined to the periphery of the window. . . The total heat developed in the dielectric element is always greater than that due to its dielectric loss factor alone. The additional heat is attributed partly to an electronic discharge and partly to the seal area losses. At high powers, electronic discharge is the predominant heat-producing mechanism and involves secondary electron resonance (multipactor) at the dielectric surface.

2-6. PHASE VI - LIFE TESTING

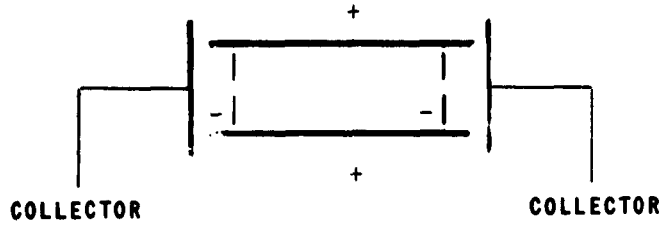
A sealed-off nonpumping ion gage is a desirable vehicle for evaluating metal-to-ceramic seals. The requirements to detect minor variations in pressure over periods of time for the extended-life and environmental tests required degrees of sensitivity which cannot be obtained from the use of helium mass spectrometers. A sealed-off pressure-measuring gage could, upon activation at predetermined intervals, indicate the gradual and progressive degradation of the vacuum attributable to minute leaks. Normal ionization gages contain filaments which, when initially heated, emit large quantities of gas relative to the gas already in evacuated chambers. However, these gages have a pumping action which, in a finite length of time, restores the chamber to a pressure level equal to and eventually better than that which originally existed. Cold cathode gages have been designed to eliminate the problem of gas emission by the filaments. The need for a nonpumping ionization gage suggested a redesign of a Penning gage, specifically to meet the objectives of this program.

A Penning cell consists of electrodes in the following configuration:



Electrons emitted from the cold cathode, or found in the volume, are deflected by means of a magnetic field so as to move in a tight circle perpendicular to that magnetic field. The electric-field configuration causes the electrons to move with a periodic motion between the cathodes. The resulting motion is a tight helix the axis of which is parallel to the direction of the magnetic field. As the electrons spiral, they collide with neutral gas atoms, forming positive ions and electrons. The ions will travel directly to the cathode to be collected. The newly formed electrons continue to spiral, but their number is controlled by space-charge limitation. Scattering by collisions allows the electrons which are in excess of the limit to be collected by the anode. The magnitude of the total current, which is the sum of the positive ion current to the cathode and the electron current from the same electrode, is used as a measure of the gas pressure present.

Pumping in a Penning cell will occur when gas ions react with the cathode to form stable compounds, or when gas ions are driven into the collecting electrode with moderately high energy. The essential feature of the nonpumping gage is the collector shown in the following sketch:



Instead of using the cathode as a collector, a special central-hole cathode is used with a separate collector. Most ions pass through the hole because of the field configuration. A retarding voltage between the cathode and the collector reduces the kinetic energy of the ions so that ions will not be pumped by being driven into the cathode. The collector is goldplated to eliminate chemical reactions on the surface. Therefore, little pumping will occur on the collector surface because of chemical reactions or surface bombardment.

Initial attempts to fabricate this gage from ceramic samples available from production tubes resulted in four assemblies which contained leaks, as determined by the helium mass spectrometer. When the ceramic materials ordered for this program arrived, several more gages were constructed. These were then placed on the r-f mass spectrometer high-vacuum station shown in figure 11. Initially, the resistance across each ceramic ring was greater than 10^{12} ohms, but after a few minutes of operation the leakage resistance dropped to approximately 10^{5} ohms, resulting in a high leakage current which masked any of the positive ion current. Dissection of the failed gages revealed that the goldplating on the copper had diffused into the body of the assemblies, exposing copper surfaces which were then active and could chemically absorb gases or react. In addition, a sputtering or discharge had occurred in the active anode section of the gage, sputtering metal across the ceramic insulators on the internal vacuum portion of the ceramics. These initial gages were brazed with copper-silver eutectic and a chemical analysis of the deposits on the ceramic revealed silver, gold, and copper in that decreasing order of magnitude.

A copper-gold eutectic brazing material was decided upon to minimize the easily sputtered silver. Several additional gages were constructed employing this brazing material. In addition, a design modification (shown in figure 12) enabled the copper assemblies to be brazed to ceramic members without gold-plating. After the whole structure had been brazed and leak checked, the samples were sealed to a closed circulating system containing electroless gold solution and were plated to a minimum thickness of 0.0002 inch of gold on all internal metal surfaces. Thorough rinsing and drying of the plated gages produced units which had completely inert inner surfaces, and which would be nonpumping. One end was then pinched off, with the resultant assembly being physically similar to the original design.

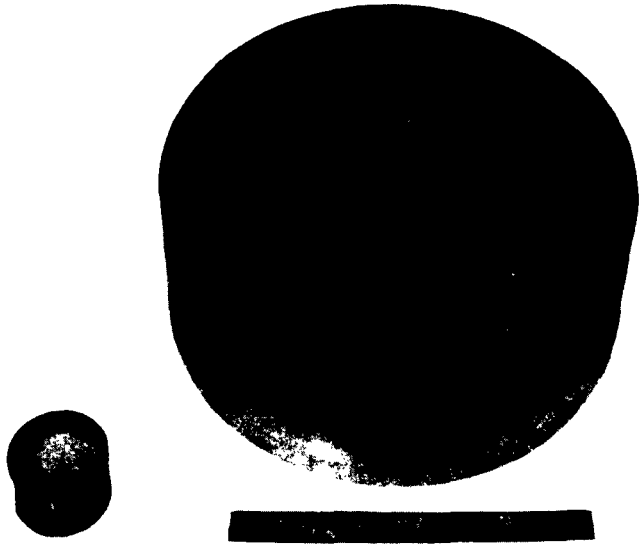


FIGURE 10. TYPICAL DISC WINDOWS

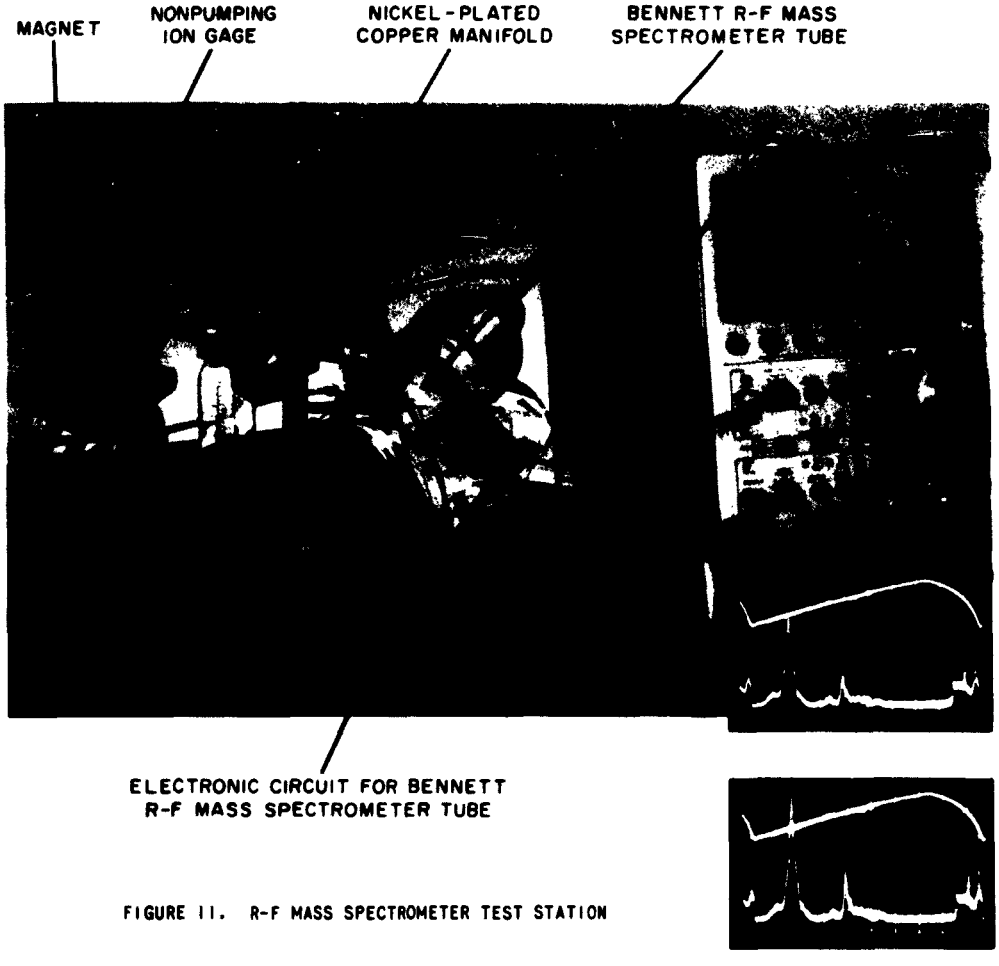


FIGURE 11. R-F MASS SPECTROMETER TEST STATION

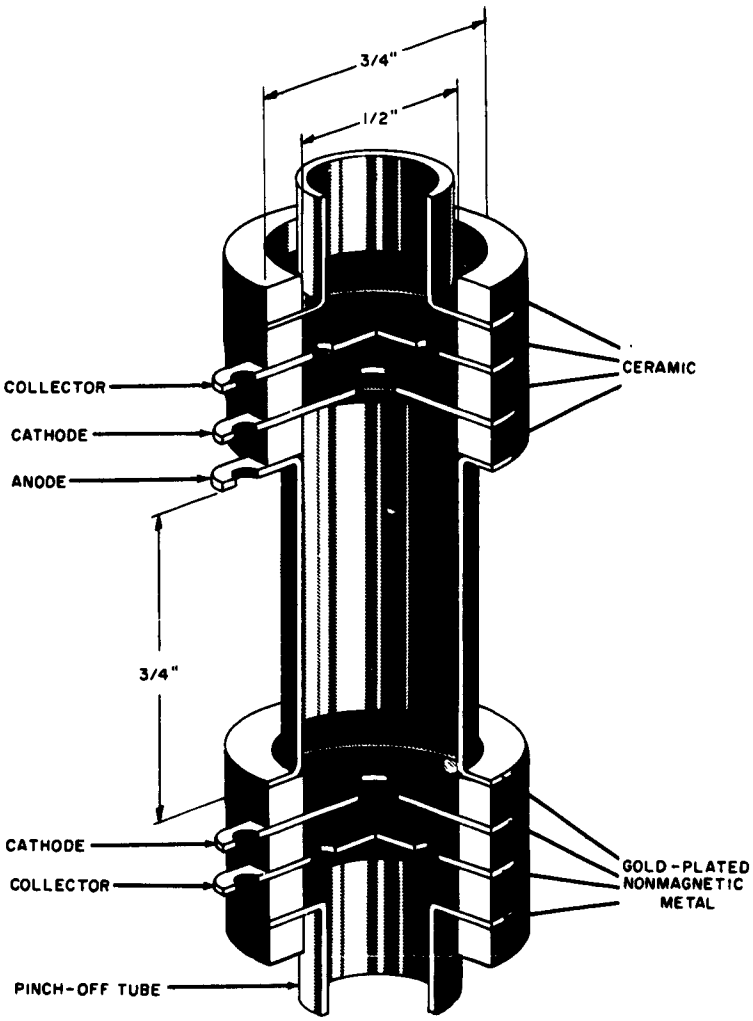


FIGURE 12. MODIFIED NONPUMPING ION GAGE

The gages were then attached to the r-f mass spectrometer station and a range of voltages from 100 to 2000 volts and magnetic fields from 700 to 1500 gauss were applied to find a combination which would be sensitive to gas pressures within the device. After considerable manipulations with these parameters, the gages were observed to be sensitive to gas pressures. A calibration curve for a gage is illustrated in figure 13. The gage functioned 2 weeks before the end of the program; thus, no time was available for further work.

2-7. PHASE VII - LEAK PATH STUDY

A total of 141 seals was employed in the development of leak path study techniques and 46 seals were used in the analysis of leak path mechanisms. These included window, coaxial antenna, and other compression seals. This technique to analyze the seal failures is not a routine production operation which could be employed for everyday failure analysis and quality assurance programs; instead, it is a precise and time-consuming operation to classify leak mechanisms in order to understand more fully the type of failures associated with the particular ceramic, metal, and braze combination.

The total accumulated percentages of various causes attributable to failures in the field are shown in figure 14. The most outstanding failure, accounting for almost half, is an insufficient or porous braze. Any of several mechanisms can cause a porous braze. The carbon content of the brazing material, particularly copper-silver eutectic, can cause carbon precipitation and gas bubbles. Also, poor wetting of oxidized surfaces, with resultant occlusions of nonreducible oxides (such as manganese dioxide and silica), and disturbance of the braze during its freezing cycle can affect the structure of the metal.

The porous ceramic can be controlled by destructive statistical sample testing of representative pieces of the ceramic, employing dark-field illumination of the stained and decomposed sections (see figure 15). Porous metallizing can be caused by long time intervals under adverse conditions between sintering and electroplating, enabling the refractory oxide of manganese to be formed, which then prevents the electroplating from completely filling the pores of the metallizing.

Braze-metallize interface failures occur when the plated layer is too thick, setting up stresses in the bond between

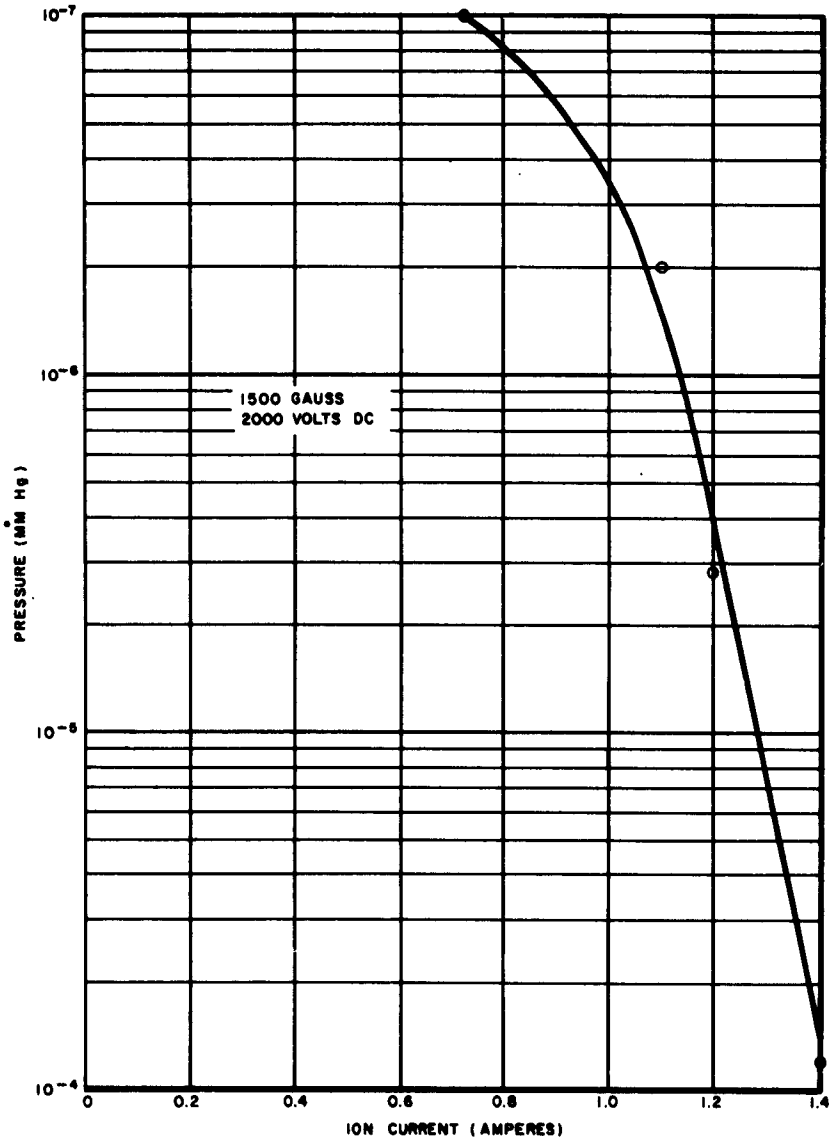


FIGURE 13. CALIBRATION CURVE FOR NONPUMPING ION GAGE

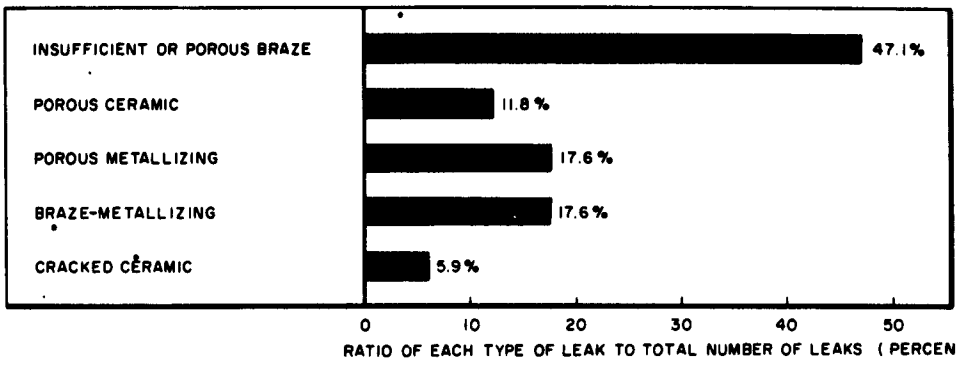


FIGURE 14. SUMMARY OF LEAK PATH MECHANISMS

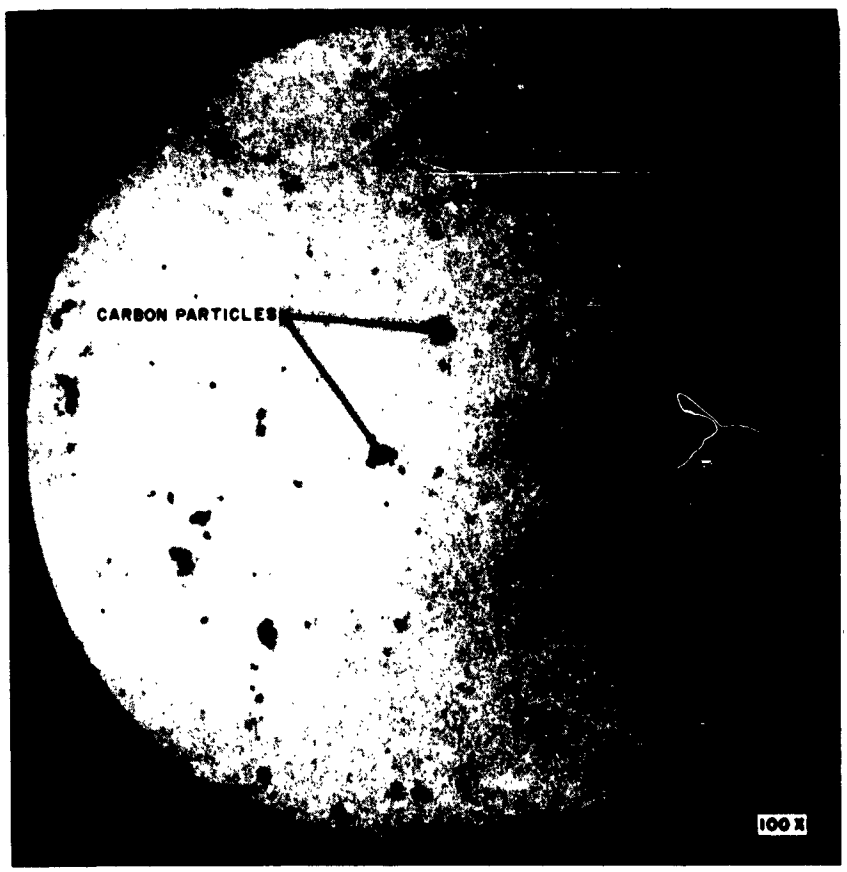


FIGURE 15. DARK-FIELD ILLUMINATED CERAMIC SAMPLE

the metallizing and the plating. Occluded plating salts in the porous metallizing, if allowed to stand too long after plating, will corrode much of the substrate material beneath the electroplated layer, weakening this interface. Another common cause of braze-metallize failure is the oversintering of the metallizing, allowing the glassy phase of the ceramic to impregnate and coat the metallizing layer. Subsequent electroplating deposited upon this conductive layer will be loosely adherent, in some cases slipping off during the removal of the samples from the plating rack. If the material adheres enough to be assembled for brazing, the brazing operation dissolves the electroplated layer and will not wet the glass-impregnated metallizing; thus, an extremely weak bond of a mechanical nature is formed which will fail upon subsequent stressing. Cracked ceramics appear at high stress points and can be minimized by proper chamfering or seal design.

The difficulties attendant with discovering and solving the origin and path of the particular leak (accomplished by multiple polishings and operations) require extreme patience. Some leaks in the study were followed from surface to surface, and indicated several mechanisms--such as cracked ceramic, porous braze, and porous ceramic. In these instances, subjective evaluations, based on experience in observing many leak paths, were employed which may color the results somewhat depending upon the investigators. Figures 16 through 21 indicate typical samples sectioned and polished after the staining operation.

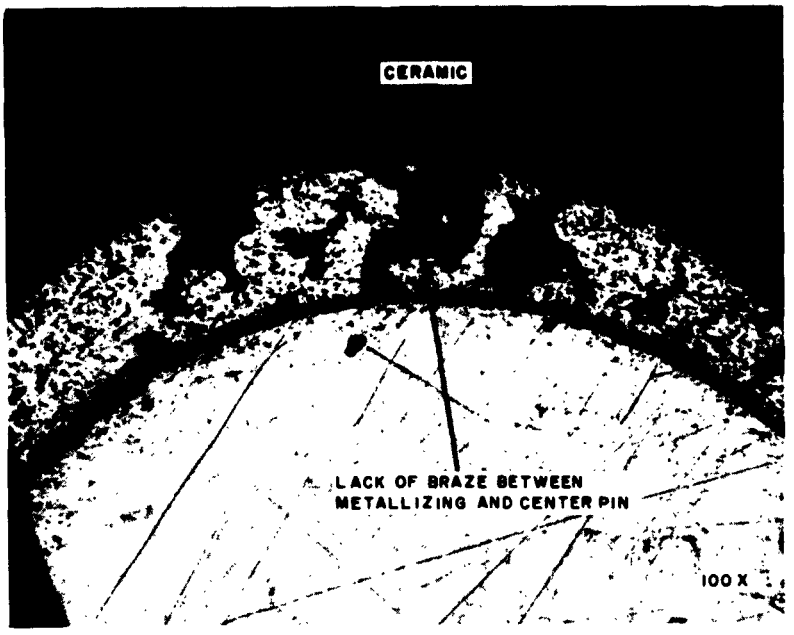


FIGURE 16. CROSS SECTION OF CENTER PIN AND BRAZE, SHOWING LACK OF BRAZE

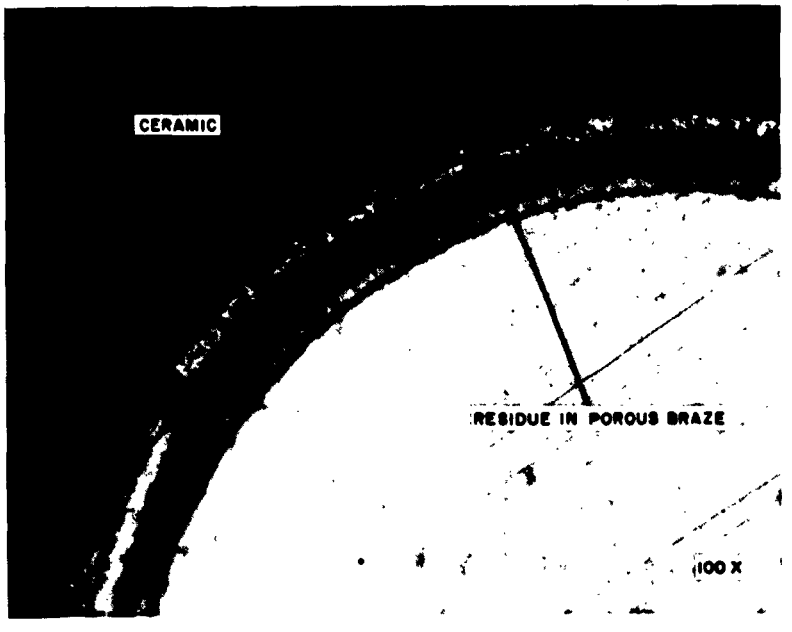


FIGURE 17. CROSS SECTION OF CENTER PIN AND BRAZE, SHOWING RESIDUE IN POROUS BRAZE

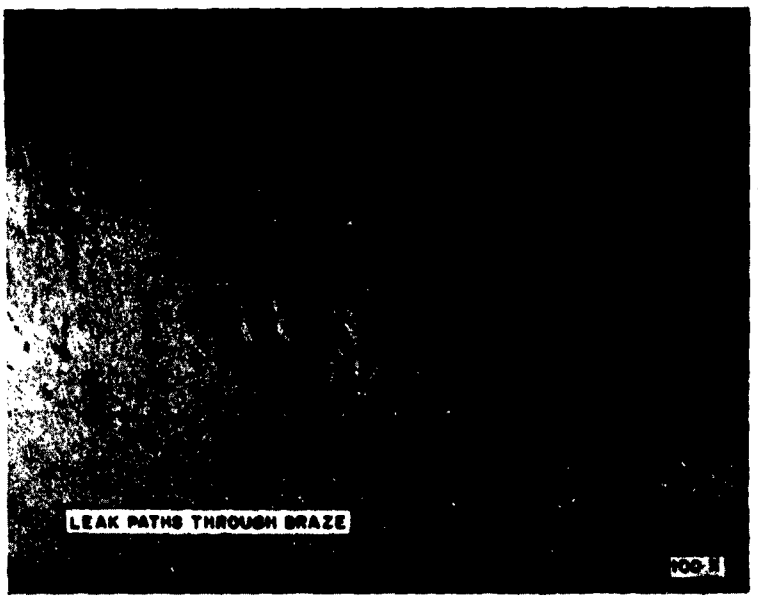


FIGURE 18. POROUS BRAZE ON OD SEAL

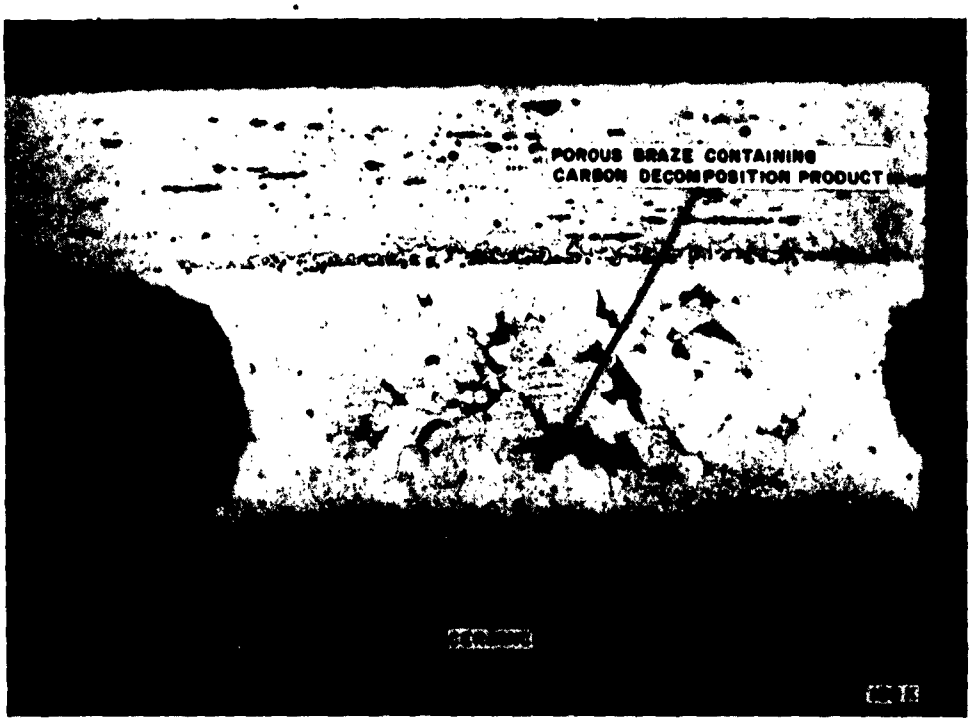


FIGURE 19. POROUS BRAZE CONTAINING CARBON DECOMPOSITION PRODUCT ON ID SEAL

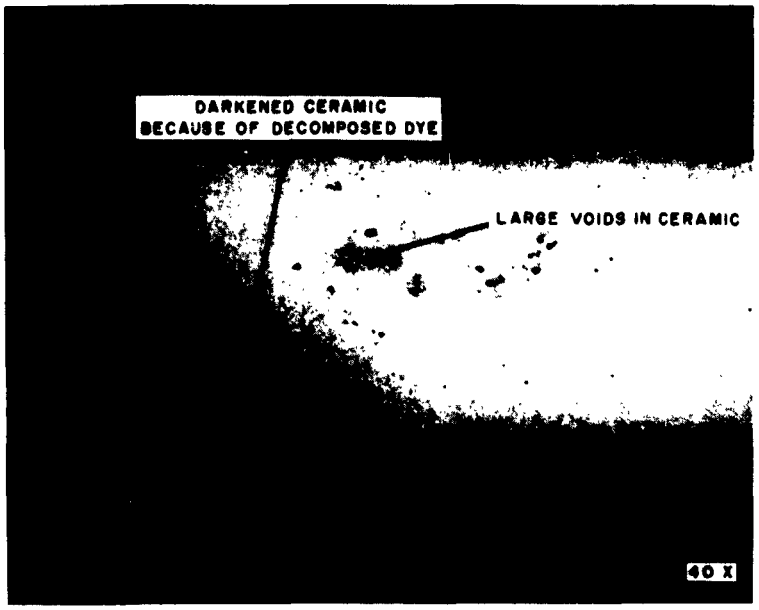


FIGURE 20. POROUS CERAMIC LONGITUDINAL SECTION

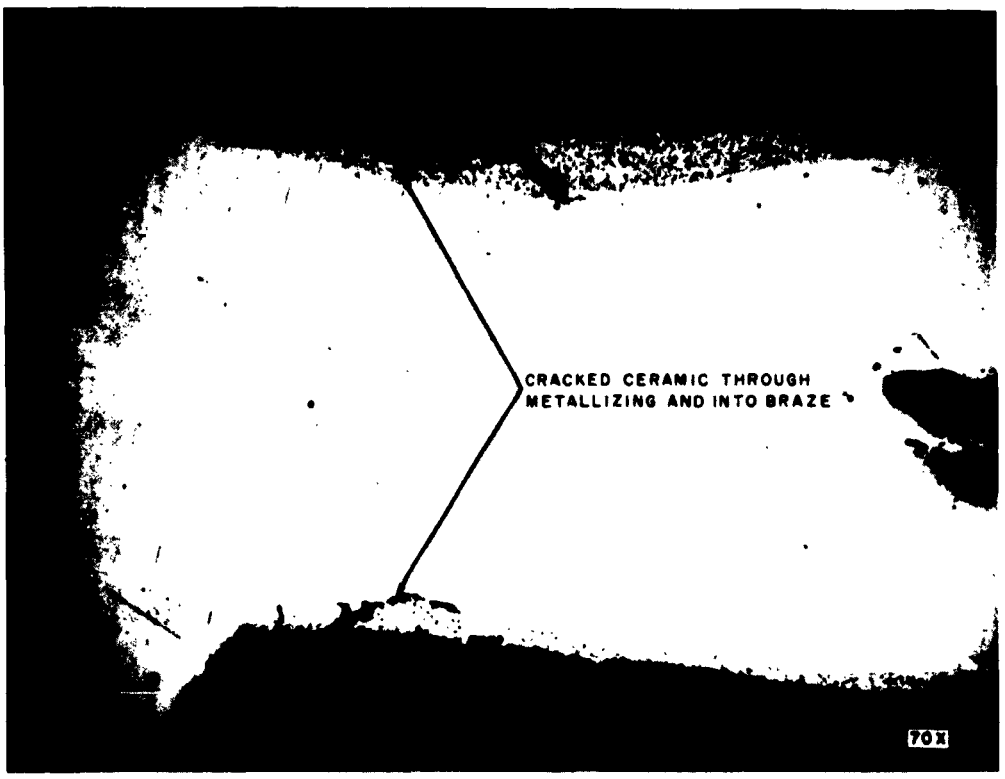


FIGURE 21. LEAK PATH BETWEEN METALLIZED LAYER AND CERAMIC

SECTION III

CONCLUSIONS

The results of extensive experimental investigations under precise laboratory controls revealed serious shortcomings in the test vehicle chosen for the evaluation of the variables examined. The residual error, caused by uncontrolled and/or unknown variables acting independently of the scheduled tests, masked the results to such an extent that only general trends could be discerned in the over-all results. Interactions of the ceramics and the various mixes appeared to contribute to the greatest variations, with the seal strength generally being inversely proportional to the percent alumina content of the ceramic body.

A nonpumping ion gage was constructed which enabled the measurement of gas pressures in closed systems without altering the pressure. The size and sensitivity of the gage suggest several interesting uses in which pressure measurements or leak monitoring is required.

A technique for tracing leak paths was developed for the purpose of classifying leak path mechanisms. Data were compiled indicating the relative populations of causes of failure in hermetic seals. Insufficient or porous brazes appeared to be the predominant mechanism.

SECTION IV

RECOMMENDATIONS

The high residual error of the modified ASTM test vehicle suggests an investigation leading to a test vehicle which would accurately indicate those variables under test. It is believed that a compression-seal assembly resembling a ceramic r-f window would fulfill this requirement. This program indicated that the residual error of the compression-seal vehicle was considerably (approximately 60 percent) lower. The determination of the dimensional parameters and the correlation with ASTM data should be made to relate all past data to the new test method.

Refinement of the nonpumping gage would lead to an inexpensive vacuum test gage. It would be useful in nuclear radiation studies, life lead tests, and a variety of other pressure-measuring applications.

A comparison of several ceramic bodies (from various manufacturers) with the same percent alumina content would indicate the variations which can occur due to the binder or glassy phase compositional differences. This would enable a more detailed analysis to be made of the role these binder components contribute to the sealability of the ceramics.

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8. W. D. Kingery, Introduction to Ceramics, (New York, 1960) p. 353 ff.
9. Unpublished results of investigations conducted by Sperry Gyroscope Company.
10. E. S. Pearson, et al; "Tables of Percentage Points of the Inverted Beta (F) Distribution," Brimetrika, Vol. 33 (1943) p. 73.
11. D. B. Churchill, High-Power Waveguide Windows, presented at the High-Power Tube Symposium, USASRDL, Fort Monmouth, 25-26 September 1962.

TABLE 1

EXPERIMENT 1 - DESIGN

Sulfamate Nickel								Ammonium Nickel							
0.0002 inch				0.0004 inch				0.0002 inch				0.0004 inch			
AL300		AD94		AL300		AD94		AL300		AD94		AL300		AD94	
1*	2**	1	2	1	2	1	2	1	2	1	2	1	2	1	2
1-1	2	33	34	3	4	35	36	5	6	37	38	7	8	39	40
9	10	41	42	11	12	43	44	13	14	45	46	15	16	47	48
17	18	49	50	19	20	51	52	21	22	53	54	23	24	55	56
25	26	57	58	27	28	59	60	29	30	61	62	31	32	63	64

Metallize: All on 1 day. Mix 65B. 64 halves AL300; 64 halves AD94; 1 coat.

Sinter:
1. First row across all A and B's (32 halves)
2. Second row across all A and B's (32 halves)
3. Third row across all A and B's (32 halves)
4. Fourth row across all A and B's (32 halves)

Plating: 4 different conditions sent in 4 separate times (8 halves at a time)

Brazing: 4 separate halves (8 at a time) of each of 2 brazing conditions

*Brazing schedule 1: 10-15-10-10

**Brazing schedule 2: 15-15-15-15

TABLE 2

EXPERIMENT 1 - RAW DATA

Sample	psi	Sample	psi	Sample	psi	Sample	psi	Grand Total $\bar{x}$ = 9001 s = 1998 cv(%) = 22
40	10560	48	7700	56	10780	64	7480	$\bar{x}$ = 9130 s = 1783 cv(%) = 20
39	12870	47	10945	55	9625	63	8910	$\bar{x}$ = 10587 s = 1740 cv(%) = 16
8	9020	16	6765	24	6985	32	4785	$\bar{x}$ = 6889 s = 1731 cv(%) = 25
7	11220	15	10835	23	9625	31	4730	$\bar{x}$ = 9103 s = 2993 cv(%) = 33
38	8965	46	10175	54	10780	62	7700	$\bar{x}$ = 9405 s = 1364 cv(%) = 14
37	11715	45	8360	53	9405	61	6490	$\bar{x}$ = 8993 s = 2179 cv(%) = 24
6	6820	14	8690	22	9020	30	5335	$\bar{x}$ = 7466 s = 1720 cv(%) = 23

TABLE 2

EXPERIMENT 1 - RAW DATA (Cont)

Sample	psi	Sample	psi	Sample	psi	Sample	psi	Grand Total $\bar{x}$ = 9001 s = 1998 cv(%) = 22
5	9625	13	9020	21	6215	29	8085	$\bar{x}$ = 8236 s = 1489 cv(%) = 18
36	11495	44	6325	52	10725	60	14355	$\bar{x}$ = 10725 s = 3323 cv(%) = 31
35	10890	43	11495	51	7480	59	9790	$\bar{x}$ = 9914 s = 1769 cv(%) = 18
4	8580	12	9185	20	8360	28	7095	$\bar{x}$ = 8305 s = 879 cv(%) = 11
3	10230	11	8360	19	6380	27	8195	$\bar{x}$ = 8291 s = 1573 cv(%) = 19
34	10340	42	8360	50	9845	58	9460	$\bar{x}$ = 9501 s = 842 cv(%) = 9
33	8855	41	10495	49	6655	51	10450	$\bar{x}$ = 9114 s = 1808 cv(%) = 20

TABLE 2

EXPERIMENT 1 - RAW DATA (Cont)

Sample	psi	Sample	psi	Sample	psi	Sample	psi	Grand Total $\bar{x}$ = 9001 s = 1998 cv(%) = 22
2	13035	10	5500	18	7755	26	10560	$\bar{x}$ = 9213 s = 3283 cv(%) = 36
1	9295	10	9955	18	9900	26	7425	$\bar{x}$ = 9144 s = 1184 cv(%) = 13
	$\bar{x}$ = 10220 s = 1648 cv(%) = 16		$\bar{x}$ = 8885 s = 1735 cv(%) = 20		$\bar{x}$ = 8721 s = 1612 cv(%) = 18		$\bar{x}$ = 8178 s = 2457 cv(%) = 30	

TABLE 3

EXPERIMENT 2 - DESIGN

AL300																	
Roller						Spray						Paint					
65B-2*		65B-5		65B-10		65B-2		65B-5		65B-10		65B-2		65B-5		65B-10	
1**	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
1	13	25	37	49	61	73	85	97	109	121	133	145	157	169	181	193	205
2	14	26	38	50	62	74	86	98	110	122	134	146	158	170	182	194	206
3	15	27	39	51	63	75	87	99	111	123	135	147	159	171	183	195	207
4	16	28	40	52	64	76	88	100	112	124	136	148	160	172	184	196	208
5	17	29	41	53	65	77	89	101	113	125	137	149	161	173	185	197	209
6	18	30	42	54	66	78	90	102	114	126	138	150	162	174	186	198	210

AD94																	
Roller						Spray						Paint					
65B-2*		65B-5		65B-10		65B-2		65B-5		65B-10		65B-2		65B-5		65B-10	
1**	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
7	19	31	43	55	67	79	91	103	115	127	139	151	163	175	187	199	211
8	20	32	44	56	68	80	92	104	116	128	140	152	164	176	188	200	212
9	21	33	45	57	69	81	93	105	117	129	141	153	165	177	189	201	213
10	22	34	46	58	70	82	94	106	118	130	142	154	166	178	190	202	214
11	23	35	47	59	71	83	95	107	119	131	143	155	167	179	191	203	215
12	24	36	48	60	72	84	96	108	120	132	144	156	168	180	192	204	216

*This row across indicates ceramic application mix number.

**This row across indicates the number of coats applied.

TABLE 4

EXPERIMENT 2 - RAW DATA

Ceramic	AL300																	
Application	Roller						Spray						Paint					
Mix No.	65B-2		65B-5		65B-10		65B-2		65B-5		65B-10		65B-2		65B-5		65B-10	
No. of Coats	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
Replicate 1*	1	13	25	37	49	61	73	85	97	109	121	133	145	157	169	181	193	205
Replicate 2*	2	14	26	38	50	62	74	86	98	110	122	134	146	158	170	182	194	206
Replicate 3*	3	15	27	39	51	63	75	87	99	111	123	135	147	159	171	183	195	207
Replicate 1-psi	9955	15070	11330	13750	7810	17930	14300	11385	13420	10230	13915	11550	13310	7425	12980	14080	7700	*8525
Replicate 2-psi	8800	9570	5500	13035	8965	12100	13530	13750	12650	7425	11330	10450	10230	10010	5775	8470	6600	10670
Replicate 3-psi	14300	11605	7810	12320	9680	11550	11440	6710	7700	5885	12760	7040	10010	11055	11000	10065	6600	8305

Ceramic	AD94																	
Application	Roller						Spray						Paint					
Mix No.	65B-2		65B-5		65B-10		65B-2		65B-5		65B-10		65B-2		65B-5		65B-10	
No. of Coats	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
Replicate 1*	7	19	31	43	55	67	79	91	103	115	127	139	151	163	175	187	199	211
Replicate 2*	8	20	32	44	56	68	80	92	104	116	128	140	152	164	176	188	200	212
Replicate 3*	9	21	33	45	57	69	81	93	105	117	129	141	153	165	177	189	201	213
Replicate 1-psi	8250	11550	11220	11605	10065	11770	8690	11385	8635	15950	11000	16280	12650	13750	17710	16720	11000	10450
Replicate 2-psi	11110	5720	8085	11440	6160	11825	11275	16170	15400	12540	7645	12650	6710	7590	6160	9900	16335	10450
Replicate 3-psi	9955	12320	14080	10230	11550	11550	11880	17545	12045	15620	8250	14905	8855	9350	12265	12650	10285	17435

*Figures to right are code numbers of samples

TABLE 5

EXPERIMENT 2 - ANALYSIS OF VARIANCE FOR ONE LAYER

No. of Averages	No. of Tensile Values in Each Average	Source of Variation	Sums of Squares	Degrees of Freedom	Mean Square (Variance)	F Ratio
2	27	Ceramic (C)	1039131	(C-1) = 1	1039131	
3	18	Application methods (A)	10586380	(A-1) = 2	5293190	
3	18	Ball milling time (B)	27722896	(B-1) = 2	13861448	1.798
6	4	CxB	33641545	(C-1)(B-1) = 2	16820773	2.182
6	9	CxA	33341506	(C-1)(A-1) = 2	16670753	2.162
9	6	BxA	3087627	(B-1)(A-1) = 4	771907	
18	3	CxBxA	36115966	(C-1)(B-1)(A-1) = 4	9028991	1.171
3	18	Replicates (R) (Error)	277543749	(CxAxB)(R-1) = 36	7709549	
1	54	Total	423078800	(CxAxBxR-1) = 53		
51						

TABLE 6

EXPERIMENT 2 - ANALYSIS OF VARIANCE FOR TWO LAYERS

No. of Averages	No. of Tensile Values in Each Average	Source of Variation	Sums of Squares	Degrees of Freedom	Mean Square (Variance)	F Ratio
2	27	Ceramic (C)	45173560	(C-1) = 1	45173560	6.424*
3	18	Application methods (A)	5427192	(A-1) = 2	2713596	
3	18	Ball milling time (B)	13980881	(B-1) = 2	2990441	
6	9	CxB	5028329	(C-1)(B-1) = 2	2514165	
6	9	CxA	127580156	(C-1)(A-1) = 2	63790078	9.071**
9	6	BxA	25288319	(B-1)(A-1) = 4	6322079	
18	3	CxBxA	6205516	(C-1)(B-1)(A-1) = 4	1551379	
3	18	Replication (R) (Error)	253160234	(CxAxB)(R-1) = 36	7032239	
1	54	Total	481844187	(CxAxBxR-1) = 53		
51						

*5-percent level-of-significance
**0.1-percent level-of-significance

TABLE 7

AVERAGE STRENGTH OF METALLIZING BOND

Ceramic	Experiment 1	Experiment 2
AL300	8331 psi	10544 psi
AD94	9671 psi	11604 psi

TABLE 8

EXPERIMENT 4 - DESIGN*

Sinter 1									
	Plate 1			Plate 2			Plate 3		
Braze	B_1	B_2	B_3	B_1	B_2	B_3	B_1	B_2	B_3
Sample No.	1	2	3	4	5	6	7	8	9
psi	5830	8470	8635	7480	8250	5225	9845	7315	10065
Sinter 2									
	Plate 1			Plate 2			Plate 3		
Braze	B_1	B_2	B_3	B_1	B_2	B_3	B_1	B_2	B_3
Sample No.	10	11	12	13	14	15	16	17	18
psi	6710	6270	7040	7150	8085	10560	7865	5885	7425
Sinter 3									
	Plate 1			Plate 2			Plate 3		
Braze	B_1	B_2	B_3	B_1	B_2	B_3	B_1	B_2	B_3
Sample No.	19	20	21	22	23	24	25	26	27
psi	5280	6380	8030	8195	6600	8470	12650	5060	5610

*All samples were AL300 ceramic, single-layer metallized; average tensile strength 7570 ± 1780 psi; coefficient of variation 23.5 percent

TABLE 9

EXPERIMENT 4 - TIME SCHEDULE

Day	Metallizing	Sintering	Plating	Brazing	Tensile Test
1	Metallize 54 halves AL300 4-1; 4-2 4-27** Both A and B 54*	Sinter 1, 2, 3, 4, 5, 6, 7, 8, 9 Both A and B 18*	Plate 1, 2, 3A and B 6*	Braze 1 A and B 2*	1
2		Sinter 10, 11, 12, 13, 14, 15, 16, 17, 18, Both A and B 18*	Plate 4, 5, 6, 10, 11, 12, A and B 12*	Braze 2, 4, 10 A and B 6*	3
3		Sinter 19, 20, 21, 22, 23, 24, 25, 26, 27 A and B 18*	Plate 7, 8, 9, 13, 14, 15, 19, 20, 21 A and B 18*	Braze 3, 5, 7, 11, 13, 19 A and B 12*	6
4			Plate 16, 17, 18, 22, 24, A and B 12*	Braze 6, 8, 12, 14, 16, 20, 22 A and B 14*	7
5			Plate 25, 26, 27 A and B 6*	Braze 9, 15, 17, 21, 23, 25 A and B 12*	6
6				Braze 18, 24, 26 A and B 6*	3
7				Braze 27 A and B 2*	1

*Number of halves used in this experiment

**Sample number prefix 4 dropped from sample number in sintering, plating, and brazing columns

TABLE 10

EXPERIMENT 3 - DESIGN

Sintering Temperature (°C) and Time Cycle (hours)								
1425			1500			1575		
4*	6*	8*	4	6	8	4	6	8
1*	2	3	4	5	6	7	8	9
10	11	12	13	14	15	16	17	18
19	20	21	22	23	24	25	26	27
28	29	30	31	32	33	34	35	36

*Each numbered box represents 15 combinations of ceramics and mixes.
Each row across represents a replicate (one sample at every condition).

TABLE 11

EXPERIMENT 3 - METALLIZING MIX COMPOSITIONS

Sample	Ceramic	Mix	Weight (grams)					
			Mo	Ti	$LiMoO_3$	CaO_2	SiO_2	Mn
1	AD94	65A	291	9.0				
2	AD94	65B	292.5	7.5				
3	AD94	65C	294	6.0				
4	AD94	91A	285		15			
5	AD94	91B	270		30			
6	AD94	91C	240		60			
7	AL300	72A	240			73.6		
8	AL300	72B	276.8			36.8		
9	AL300	72C	295.2			18.4		
10	AD995	50A	255				48	22
11	AD995	50B	290				24	11
12	AD995	50C	307.5				12	5.5
13	AL300	65A	291	9.0				
14	AL300	65B	292.5	7.5				
15	AL300	65C	294	6.0				
16	AD94	Sperry	240					60

TABLE 12

EXPERIMENT 3 - RAW DATA FOR AD94*

Time Cycle (hours)	Mix	Sintering Temperature °C		
		1425	1500	1575
4	1	9103	10931	8113
	2	9900	12238	9721
	3	9515	13791	10120
	4	9116	11495	10423
	5	8896	11083	10684
	6	8236	10285	9639
	16	8278	10849	10931
	Avg	9006	11525	9947
6	1	10395	8553	10216
	2	9859	12416	11234
	3	10588	13076	10093
	4	10051	10368	9886
	5	8828	11591	10821
	6	7755	9983	10423
	16	8978	10588	10285
	Avg	9493	10939	10423
8	1	10835	9845	10780
	2	10808	10601	7865
	3	11000	11303	12843
	4	10835	9804	10739
	5	9556	10093	11083
	6	9955	9350	10079
	16	9501	8030	9446
	Avg	10356	9861	10405

*Values in psi

TABLE 13

EXPERIMENT 3 - RAW DATA FOR AL300*

Time Cycle (hours)	Mix	Sintering Temperature °C		
		1425	1500	1575
4	7	6985	13145	10629
	8	7274	10216	10945
	9	7604	12444	10203
	13	7054	10835	11069
	14	5940	9543	10698
	15	5803	10423	11949
	Avg	6777	11101	10916
6	7	3547	10093	10533
	8	3616	11358	11358
	9	4991	9543	11138
	13	6573	11083	12691
	14	6064	10216	11193
	15	5569	11935	7315
	Avg	5060	10705	10705
8	7	9103	8814	10450
	8	8718	9941	7796
	9	8236	6545	9488
	13	7178	7989	8098
	14	3699	9941	6848
	15	6944	8759	9653
	Avg	7313	8665	8722

*Values in psi

TABLE 14

EXPERIMENT 3 - RAW DATA FOR AD995*

Time Cycle (hours)	Mix	Sintering Temperature °C		
		1425	1500	1575
4	10	6861	9941	5734
	11	5321	4015	6311
	12	5308	4304	3108
	Avg	5830	6087	5051
6	10	5431	8566	6353
	11	5899	5693	6174
	12	3878	4235	4868
	Avg	5069	6164	5798
8	10	7356	6834	7453
	11	7026	4826	6325
	12	4153	2819	4754
	Avg	6178	4826	6177

*Values in psi

Ceramic-Mix Sintering Temperature (°C)	
Time Cycle (hours)	
1	Avg. CV(%)
2	Avg. CV(%)
3	Avg. CV(%)
4	Avg. CV(%)
5	Avg. CV(%)
6	Avg. CV(%)
7	Avg. CV(%)
8	Avg. CV(%)
9	Avg. CV(%)
10	Avg. CV(%)
11	Avg. CV(%)
12	Avg. CV(%)
13	Avg. CV(%)
14	Avg. CV(%)
15	Avg. CV(%)
16	Avg. CV(%)

TABLE 15

EXPERIMENT 3 - SPECIFIC TEMPERATURE AND TIME AVERAGES FOR EACH CERAMIC-MIX COMBINATION

Ceramic-Mix Sintering Temperature (°C)		1425			1500			1575		
Time Cycle (hours)		4	6	8	4	6	8	4	6	8
1	Avg.	9103	10395	10835	10931	8553	9845	8113	10216	10780
	CV(%)	4	13	20	9	45	29	13	18	22
2	Avg.	9900	9859	10808	12238	12416	10601	9721	11234	7865
	CV(%)	27	11	19	22	16	13	27	26	55
3	Avg.	9515	10588	11000	13791	13076	11303	10120	10093	12843
	CV(%)	15	11	28	16	24	19	16	35	16
4	Avg.	9116	10051	10835	11495	10368	9804	10423	9886	10739
	CV(%)	13	17	19	20	19	25	14	15	12
5	Avg.	8896	8828	9556	11083	11591	10093	10684	10821	11083
	CV(%)	12	28	15	13	11	14	15	5	21
6	Avg.	8236	7755	9955	10285	9983	9350	9639	10423	10079
	CV(%)	12	26	6	16	7	19	12	11	21
7	Avg.	6985	3548	9103	13145	10093	8814	10629	10533	10450
	CV(%)	77	100	72	23	44	13	29	18	19
8	Avg.	7274	3616	8718	10216	11358	9914	10945	11358	7796
	CV(%)	73	56	33	40	28	16	29	38	29
9	Avg.	7604	4991	8236	12444	9543	6545	10203	11138	9488
	CV(%)	64	40	56	22	28	12	17	15	9
10	Avg.	6861	5431	7356	9941	8566	6834	5734	6353	7453
	CV(%)	32	32	37	22	30	12	47	25	26
11	Avg.	5321	5899	7026	4015	5693	4826	6311	6174	6325
	CV(%)	21	20	9	27	68	19	20	25	30
12	Avg.	5308	3878	4153	4304	4235	2819	3108	4868	4754
	CV(%)	20	68	43	32	20	52	46	18	13
13	Avg.	7054	6573	7178	10835	11083	7989	11069	12691	8098
	CV(%)	40	31	56	24	28	32	27	6	26
14	Avg.	5940	6064	3699	9543	10216	9941	10698	11193	6848
	CV(%)	90	61	34	30	22	27	37	18	53
15	Avg.	5803	5569	6944	10423	11935	8759	11949	7315	9653
	CV(%)	84	69	65	12	25	18	7	40	37
16	Avg.	8278	8979	9501	10849	10588	8030	10931	10285	9446
	CV(%)	20	16	13	18	20	18	24	15	25

TABLE 16

EXPERIMENT 3 - TEMPERATURE AND TIME AVERAGES

Sample	1. Averages (psi) of 12 samples (4 replicates at 3 time cycles) for each temperature						2. Averages (psi) of 12 sa temperatures) for each tim		
	1425°C	CV(%)	1500°C	CV(%)	1575°C	CV(%)	4 hours	CV(%)	6 H
1	10111	15	9776	28	9703	21	9382	15	9
2	10189	19	11751	18	9607	35	10619	25	11
3	10368	19	12723	20	11018	26	11142	23	11
4	10001	17	10555	20	10349	13	10344	18	10
5	9093	18	10922	13	10863	14	10220	16	10
6	8649	18	9872	14	10047	14	9386	16	93
7	6545	26	10683	32	10537	21	10253	22	8
8	6536	61	10505	27	10033	33	9478	14	8
9	6944	57	9510	34	10275	15	10083	37	8
10	6550	34	8447	27	6513	32	7512	38	6
11	6082	19	4844	47	6270	23	5213	28	5
12	4446	43	3786	35	4339	30	4239	35	4
13	6935	40	9968	29	10656	26	9652	33	10
14	5234	69	9900	24	9579	38	8726	49	9
15	6105	66	10372	23	9638	33	9391	41	8
16	8919	16	9822	22	10220	21	10019	23	9

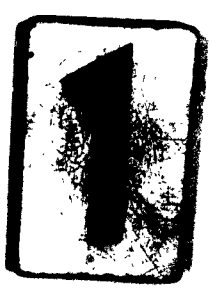

TABLE 16

EMPERATURE AND TIME AVERAGES

ycles)		2. Averages (psi) of 12 samples (4 replicates at 3 temperatures) for each time cycle						3. Averages (psi) of 36 samples (4 replicates at all temperatures and time cycles)	
:	CV(%)	4 hours	CV(%)	6 hours	CV(%)	8 hours	CV(%)	Total	CV(%)
	21	9382	15	9721	26	10486	22	9863	22
	35	10619	25	11169	20	9758	30	10516	25
	26	11142	23	11252	26	11715	20	11370	22
	13	10344	18	10101	17	10459	18	10302	17
	14	10220	16	10413	18	10243	17	10293	17
	14	9386	16	9386	18	9794	16	9523	16
	21	10253	22	8057	18	9455	39	9255	46
	33	9478	14	8777	17	8818	26	9025	42
	15	10083	37	8557	39	8089	34	8910	37
	32	7512	38	6783	34	7214	25	7170	32
	23	5213	28	5921	39	6059	25	5732	31
	30	4239	35	4326	37	3905	39	4157	36
	26	9652	33	10115	33	7791	37	9187	35
	38	8726	49	9157	37	6829	53	8238	46
	33	9391	41	8273	49	8451	39	8705	42
	21	10019	23	9950	17	8992	19	9654	20
						Total (576 Samples)		8869	36.8

TABLE 17

EXPERIMENT 3 - ORDER OF SINTERING AND REPLICATION VARIATION*

Temperature (°C)	Time Cycle (hours)	Replicate			
		1	2	3	4
1475	4	(1) 9096	(2) 9058	(3) 5510	(4) 6634
	6	(5) 8501	(6) 6253	(7) 6394	(8) 6858
	8	(9) 6469	(10) 7717	(11) 9017	(12) 10522
1500	4	(13) 11890	(14) 9756	(15) 9481	(16) 10258
	6	(17) 11134	(18) 9508	(19) 10955	(20) 8226
	8	(21) 8504	(22) 8198	(23) 8278	(24) 8893
1575	4	(25) 9897	(26) 8608	(27) 10000	(28) 9065
	6	(29) 10000	(30) 9109	(31) 10536	(32) 8999
	8	(33) 9054	(34) 7834	(35) 9903	(36) 9158

*Values in psi; averages of 16 samples at each of 9 sintering conditions and 4 replicates of each sinter. Chronological sequence of sintering samples indicated by (n).

TABLE 18

EXPERIMENT 3 - SUM OF ALL SAMPLES FOR EACH TIME AND TEMPERATURE FOR EACH CERAMIC*

Ceramic	Time Cycle (hours)	Sintering Temperature (°C)			Average
		1425	1500	1575	
AL300	4	6777	11101	10916	9597
	6	5060	10705	10705	8823
	8	7313	8865	8722	8239
	Avg	6383	10156	10120	8887
AD94	4	9006	11525	9947	10159
	6	9493	10939	10423	10285
	8	10356	9861	10405	10207
	Avg	9618	10774	10258	10217
AD995	4	5830	6087	5051	5655
	6	5069	6164	5798	5677
	8	6178	4826	6177	5726
	Avg	5693	5692	5674	5686

*Values in psi

TABLE 19

EXPERIMENT 3 - AVERAGES FOR ALL CERAMICS AT ALL SOAK AND TEMPERATURE CONDITIONS*

Time Cycle (hours)	Averages and Coefficients of Variation	Sintering Temperature(°C)			Total all Temperatures (192 Samples)
		1425	1500	1575	
4	Avg (psi)	7572	10346	9392	9104
	CV(%)	42	32	33	37
6	Avg (psi)	7001	9956	9661	8873
	CV(%)	45	33	29	38
8	Avg (psi)	8431	8468	8987	8629
	CV(%)	41	32	33	36
Total (192 Samples)	Avg (psi)	7669	9590	9347	Total 8869
	CV(%)	43	33	32	(576 37 Samples)

*16 samples replicated 4 times at each temperature and time cycle.

TABLE 20

EXPERIMENT 3 - ANALYSIS OF VARIANCE FOR AD94 (252 SAMPLES)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares (Variance)	F Ratio
7 Mixes	6	82,374,589	13,729,098	3.39*
3 Heat Cycles	2	1,968,866.8	984,433.41	-
3 Sintering Temperatures	2	3,309,085.8	1,654,542.9	-
Mixes x Heat Cycles	12	94,540,223	7,878,351.9	1.94**
Mixes x Sintering Temperatures	12	36,248,833	3,020,736.1	-
Heat Cycles x Sintering Temperatures	4	5,960,594.1	1,490,148.5	-
Mixes x Heat Cycles x Sintering Temperatures	24	143,721,570	5,988,398.8	1.47
4 Replicates	189	765,394,970	4,049,708.8	-
Total	251	1,133,518,700		

*Differences between mixes on AD94 significant at 0.1-percent level

**Differences between sintering temperatures significant at 5-percent level

TABLE 21

EXPERIMENT 3 - ANALYSIS OF VARIANCE FOR AL300 (216 SAMPLES)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares (Variance)	F Ratio
6 Mixes	5	791,207,410	158,241,480	15.41*
3 Heating Cycles	2	76,636,059.9	38,180,299	3.71**
3 Sintering Temperature	2	12,802,447	6,401,223.6	-
Mixer x Heating Cycles	10	144,348,730	14,434,873	1.40
Mixer x Sintering Temperatures	10	38,826,624	3,882,662.4	-
Heating Cycles x Sintering Temperatures	4	21,195,786	5,298,946.5	-
Mixes x Heating Cycles x Sintering Temperatures	20	131,897,000	6,594,850.3	-
4 Replicates	162	1,663,441,400	10,267,871	-
Total	215	2,880,050,000		

*Differences between mixes significant at 0.1 -percent level
**Differences between heating cycles significant at 5-percent level

TABLE 22

EXPERIMENT 3 - ANALYSIS OF VARIANCE FOR AD995 (108 SAMPLES)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares (Variance)	F Ratio
3 Mixes	2	8,753.2406	4,376.6203	-
3 Heating Cycles	2	91,161.573	45,580.786	-
3 Sintering Temperatures	2	163,493,180	81,746,591	24.93*
Mixes x Heating Cycles	4	28,980,742	7,245,185.7	2.20
Mixes x Sintering Temperatures	4	46,469,546	11,617,386	3.54**
Heating Cycles x Sintering Temperatures	4	9,211,768	2,302,942	-
Mixes x Heating Cycles x Sintering Temperatures	8	24,808,628	3,101,078.5	-
4 Replicates	81	265,579,420	3,278,758.3	-
Total	107	538,643,200		

*Differences between sintering temperatures significant at 0.1-percent level

**Differences in interaction of mixes and sintering temperatures significant at 5-percent level

TABLE 23

EXPERIMENT 5 - DESIGN

Ceramic	AL300						AD94						
Mix*	R72A			C65A			R91A			C91A			
Additive	SiO_2	Fe	Reg	SiO_2	Fe	Reg	SiO_2	Fe	Reg	SiO_2	Fe	Reg	S
Sample	1	2	3	4	5	6	7	8	9	10	11	12	1

*Letter preceding mix number indicates colloid milled (C), or roll or ball milled (R).

TABLE 24

EXPERIMENT 5 - RAW DATA*

Ceramic-Mix Combination	Greened Stainless-Steel Brazing Fixture							Graphite B		
	1	2	3	4	5	6	7**	8	9	10
1	10175	15730	16060	13200	12430	11275	4675	8058	6545	8030
2	13365	15070	8470	11165	11000	9625	8085	8525	8580	8800
3	13310	12925	8635	11880	8965	13585	6820	5940	8140	8525
4	6655	11000	16170	10395	8690	11110	5500	9405	7535	3685
5	11220	9790	17270	10450	4290	13530	5555	10560	10560	11330
6	6600	7975	12815	10340	9845	9075	5940	10890	8525	12540
7	9570	8140	10175	14520	8140	11165	3905	12485	8030	7260
8	7920	13915	11660	11825	7370	10505	5500	7755	11220	11880
9	10670	12430	11550	9570	9460	11000	5335	9735	9515	7975
10	10560	9460	12540	11275	13255	9350	4070	16005	9350	8800
11	7920	10010	11935	10505	14300	12210	4675	9790	10505	12925
12	12375	10835	9130	11550	11495	10395	3795	10175	11550	12320
13	8965	5885	5390	10395	11220	5170	3245	5060	6600	9570
14	8360	5500	10175	11605	12980	4510	2860	5885	7865	8195
15	7205	7865	9240	11000	7315	6710	2200	6545	8965	9460
16	8525	12870	9570	4785	9900	13695	2860	7700	4015	10450
17	8855	11550	7865	7425	7370	10615	1210	6985	7645	9900
18	13035	10890	17490	10065	7480	7370	3850	5335	7865	6710
Weekly Averages of all Samples	9738	10658	11452	10663	9750	10049	4448	8714	8501	9353

*Values in psi

**Inexperienced metallizing mix applicator

TABLE 23

EXPERIMENT 5 - DESIGN

AL300				AD94						AD995					
	C65A			R91A			C91A			R50A			C50A		
g	SiO_2	Fe	Reg	SiO_2	Fe	Reg	SiO_2	Fe	Reg	SiO_2	Fe	Reg	SiO_2	Fe	Reg
	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18

ndicates colloid milled (C), or roll or ball milled (R).

TABLE 24

EXPERIMENT 5 - RAW DATA*

Greened Stainless-Steel Brazing Fixture						Graphite Brazing Fixture						
2	3	4	5	6	7**	8	9	10	11**	12	13	14
15730	16060	13200	12430	11275	4675	8058	6545	8030	2530	7920	11495	8250
15070	8470	11165	11000	9625	8085	8525	8580	8800	8525	9350	11110	12320
12925	8635	11880	8965	13585	6820	5940	8140	8525	6710	8030	11990	8360
11000	16170	10395	8690	11110	5500	9405	7535	3685	9515	5940	11770	9240
9790	17270	10450	4290	13530	5555	10560	10560	11330	12100	9900	13365	13090
7975	12815	10340	9845	9075	5940	10890	8525	12540	8690	12430	10450	7920
8140	10175	14520	8140	11165	3905	12485	8030	7260	9680	10175	10835	8250
13915	11660	11825	7370	10505	5500	7755	11220	11880	8690	8580	10725	11000
12430	11550	9570	9460	11000	5335	9735	9515	7975	11770	10175	8800	8800
9460	12540	11275	13255	9350	4070	16005	9350	8800	7700	10450	6600	11440
10010	11935	10505	14300	12210	4675	9790	10505	12925	9570	8415	13530	12870
10835	9130	11550	11495	10395	3795	10175	11550	12320	7700	8470	13475	14300
5885	5390	10395	11220	5170	3245	5060	6600	9570	7975	6490	13365	8470
5500	10175	11605	12980	4510	2860	5885	7865	8195	8910	11000	15400	10285
7865	9240	11000	7315	6710	2200	6545	8965	9460	8800	10120	12650	8415
12870	9570	4785	9900	13695	2860	7700	4015	10450	4950	9735	11330	8085
11550	7865	7425	7370	10615	1210	6985	7645	9900	5225	13915	12540	11000
10890	17490	10065	7480	7370	3850	5335	7865	6710	6105	11220	11880	10780
10658	11452	10663	9750	10049	4448	8714	8501	9353	8064	9573	11739	10160

ing mix applicator

TABLE 25

EXPERIMENT 5 - ECCENTRICITY MEASUREMENTS*

Ceramic-Mix Combination	Greened Stainless-Steel Brazing Fixture							Carbon Brazing			
	1	2	3	4	5	6	7	8	9	10	11
1		39	13	11	35	13		17	11	8	19
2		37	34	45	39	20		0	17	5	6
3	31	42	45	28	22	13		8	13	3	0
4	24	28	22	33	15	20		11	7	0	14
5	21	25	14	14	14	20		10	10	10	24
6		11	29	36	9	25		0	0	12	4
7		46	36	21	32	13		9	13	28	13
8		21	12	23	26	14		20	6	4	15
9	23	14	36	7	10	18		17	0	10	6
10	41	32	20	45	13	12		8	8	13	7
11	24	0	24	38	0	40		24	0	9	10
12	13	25	13	8	12	16		11	0	12	21
13	31	21	17	41	30	27		22	7	11	20
14	34	23	24	14	20	21		4	0	20**	9
15	41	0	16	28	7	43		16	20	12	19
16	19	30	26	0	47	18		13	11	6	7
17	11	34	15	10	26	20		8	15	12	12
18	13	42	36	11	22	30		3	10	9	15
Avg	25.0	26.1	23.5	22.9	21.1	21.3		11.1	8.2	10.2	12.28

Not Available

*Measured in filar units (0.66 objective, 20 x occular, 1 filar = 0.0003 inch)
**Gap in braze

TABLE 25

EXPERIMENT 5 - ECCENTRICITY MEASUREMENTS*

ned Stainless-Steel Brazing Fixture					Carbon Brazing Fixture						
3	4	5	6	7	8	9	10	11	12	13	14
13	11	35	13		17	11	8	19	14	16	20
34	45	39	20		0	17	5	6	21	23	12
45	28	22	13		8	13	3	0	18	0	18
22	33	15	20		11	7	0	14	17	17	11
14	14	14	20		10	10	10	24	11	11	10
29	36	9	25		0	0	12	4	10	18	21
36	21	32	13		9	13	28	13	16	16	15
12	23	26	14	Not Available	20	6	4	15	35	13	14
36	7	10	18		17	0	10	6	12	13	12
20	45	13	12		8	8	13	7	8	22	19
24	38	0	40		24	0	9	10	29	14	17
13	8	12	16		11	0	12	21	12	0	21
17	41	30	27		22	7	11	20	13	14	14
24	14	20	21		4	0	20**	9	21	20	13
16	28	7	43		16	20	12	19	26	28	12
26	0	47	18		13	11	6	7	18	12	17
15	10	26	20		8	15	12	12	13	9	23
36	11	22	30		3	10	9	15	22	12	12
1 23.5	22.9	21.1	21.3		11.1	8.2	10.2	12.28	17.55	14.33	15.61

bjective, 20 x occular, 1 filar = 0.0003 inch)

TABLE 26

EXPERIMENT 5 - SUMMATION OF ALL SAMPLES FOR EACH CONDITION*

	Run	Average Tensile Strength	CV(%)	Avg. Ecc.
Metal Brazing Fixture	1	9738	23	25
	2	10658	27	26
	3	11452	30	24
	4	10664	19	23
	5	9750	27	21
	6	10050	27	21
	7**	4449	38	
Carbon Brazing Fixture	8	8714	32	11
	9	8501	22	8
	10	9353	25	10
	11**	8064	29	12
	12	9573	20	18
	13	11739	17	14
	14	10160	20	16

*14 weeks of data; values in psi.

**Inexperienced metallizing mix applicator.

TABLE 27

EXPERIMENT 5 - ANALYSIS OF VARIANCE

		Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares (Variance)	F Ratio
Metal Fixture	AL300	Milling (Colloid and Ball)	24,411,834	1	24,411,834	2.957
		Impurities	11,994,629	2	5,997,315	
		Mix x Impurities	7,633,668	2	3,816,834	
		Error	247,626,588	30	8,254,220	
	AD94	Milling	2,514,867	1	2,514,867	
		Impurities	255,276	2	127,638	
		Mix x Impurities	581,977	2	290,989	
		Error	109,146,538	30	3,638,218	
	AD994	Milling	24,775,506	1	24,775,506	3.257
		Impurities	3,862,056	2	1,931,028	
		Mix x Impurities	30,036,148	2	15,018,074	1.975
		Error	228,171,159	30	7,605,705	
Carbon Fixture	AL300	Milling	10,161,335	1	10,161,335	2.657
		Impurities	36,542,672	2	18,271,336	4.778*
		Mix x Impurities	10,547,815	2	5,273,908	1.379
		Error	114,711,025	30	3,823,701	
	AD94	Milling	21,429,183	1	21,429,183	4.856*
		Impurities	3,812,676	2	1,906,338	
		Mix x Impurities	3,614,302	2	1,807,151	
		Error	132,391,380	30	4,413,046	
	AD995	Milling	210,069	1	210,069	
		Impurities	16,279,710	2	8,139,855	1.078
		Mix x Impurities	1,452,168	2	726,084	
		Error	226,376,884	30	7,545,896	

*5-percent significance

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